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The General Secretary read the minutes of the previous meeting, which were confirmed and signed as a correct record.

The President introduced the authors and the following paper was presented in summary by Mr Holmes.

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By V. I. HOLMES † and W. K. B. MARSHALL †

SUMMARY

Steel structures, pipes, and vessels form the main capital equipment of the petroleum industry, and consequently the science of welding has become more and more an intimate and vital factor in its design and conception.

This paper seeks to review some available welding methods and to describe how they are used or can be used by the oil industry. Reference is made to pipelines, storage tanks, and to refinery equipment, and emphasis is placed upon the potentialities of new welding processes and new types of machines designed to reduce the cost of such installations.

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INTRODUCTION

IT is true to say that the petroleum industry has grown up with welding. Certainly the winning, transport, storage, and refining of petroleum on the scale that it is practised to-day would not be possible without the widespread use of welding in the fabrication of pipelines, storage tanks, and refinery and associated chemical plant, not to mention the part it plays in the building of the modern oil tanker. It is not surprising therefore that the industry has from the very start been a vigorous pioneer of welding. This has not meant the mere exploitation of existing welding methods but the development of new methods inspired by a new refreshing outlook on the potentialities of the welding processes. These new techniques have often been assimilated and applied with advantage by other branches of industry.

Against this background it is with understandable deference that the present authors offer their views on some of the newer techniques which the manufacturers of welding equipment have developed and which are now being exploited by the oil industry. At the same time the opportunity of commenting on certain traditional techniques will not be missed, as in many fields the well-tried methods still remain unchallenged.

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MATERIAL AND EQUIPMENT

Manual Welding

The two leading marks of progress in manual welding in recent years are the perfecting of the low hydrogen type electrode and the introduction of the iron powder coating.

The theory of the low hydrogen electrode has been recognized for a long time, but it is only recently that the usability and metallurgical properties have been improved to an extent that has permitted it to play a significant part. The lime–fluoride type coating suitably formulated and dried to give a low hydrogen content in the deposited metal is now the basis of all electrodes used for welding difficult steels and even mild steel, when special resistance to hot cracking is required and when particularly good notch ductility is to be achieved. This type of coating with a chromium, nickel, and molybdenum content is also used on electrodes depositing low alloy weld metal of the high tensile and creep resisting varieties.

The iron powder electrode, again, is not new, but recent advances in formulation have brought it into the field, almost as a process, to speed mass production. The rutile varieties are designed to yield depositions up to 180 per cent of the core wire weight, and

* MS received 27 February 1961.

† Rockweld Ltd.

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Against this background it is with understandable deference that the present authors offer their views on some of the newer techniques which the manufacturers of welding equipment have developed and which are now being exploited by the oil industry. At the same time the opportunity of commenting on certain traditional techniques will not be missed, as in many fields the well-tried methods still remain unchallenged.

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are used to excellent effect in fillets in the downhand and horizontal/vertical positions. Gauge for gauge these electrodes permit metal to be deposited at rates up to twice that possible with conventional types, and furthermore the presence of the iron powder gives a measure of conductivity to the flux which gives easy arc striking. The speed of welding and the easy control of the iron powder electrodes frequently make a case for manual welding, as opposed to automatic welding, when the weldment is complicated and many broken runs are involved. Table I is an interesting tabulation of comparison between typical rutile electrodes and commonly used high production rate iron powder electrodes.

TABLE I

Comparison Between Typical Standard and Iron Powder Rutile Type Electrodes—Horizontal/Vertical Fillet Weld

	Gauge	Current, amp	Fillet leg length, inches	Fillet length of 12 inches of electrode, inches	Fillet length per min, inches	Weight of weld metal per min, lb
Standard rutile	10 swg	135	$\frac{1}{4}$	8	8	0·041
Iron powder rutile	10 swg	170	$\frac{1}{4}$	12	12½	0·065
Standard rutile	8 swg	180	$\frac{1}{4}$	9	7½	0·055
Iron powder rutile	8 swg	225	$\frac{1}{4}$	14	14	0·095
Standard rutile	6 swg	225	$\frac{5}{16}$	11	8½	0·072
Iron powder rutile	6 swg	275	$\frac{5}{16}$	16	16½	0·135

Conventional Automatic Welding

The expansion of the field of automatic welding has been more spectacular than that of manual welding. The main problems of automatic welding are associated with the bringing together of electrical energy, fluxing media, and a continuous wire feed, and inevitably they have been accentuated by the higher deposition rates and the more exacting tests demanded by progress. Historically, the successful solutions have followed two distinct lines.

On the one hand is the submerged arc process utilizing bare wire and loose granulated flux enclosing the arc. Bare wire is an inexpensive raw material and has a very real attraction in the extent to which it simplifies the problem of introducing the welding current to the arc. High current densities in the wire become possible, and relatively small diameter wire fed at relatively high speed has played an important part in achieving high rates of production and controlled quality welds. A considerable amount of work has been done in the development of fluxes, which with mild steel wire can produce low alloy and hard surfacing deposits. The principal disadvantage of the submerged arc process is the inescapable fact that the arc is invisible, and hence special care must be taken in plate fit-up and machine alignment to make certain that the weld is centralized on the seam. There is also the additional complication of handling the loose flux and of recovering the surplus, and, of course, the limitation to indoor or well protected applications.

The other traditional method of automatic welding is that employing a continuous flux coated wire in coil form. This process is really an extension of manual welding in that conventional flux formulæ are used, but the additional problems of conducting the current through the flux to the core wire and of securing sufficient flexibility for coiling are encountered. Various solutions have been developed, but the most effective employs a helix of thinner wire wound round the main core and laid in such a way as to bind the flux, while at the same time providing a conducting path from the outer skin to the core. The reinforcing effect of the helix, or helices, and the manner in which it divides the mass of the flux gives incidentally the required flexibility. In spite of the fact that this process has the advantages of a visible arc and has been exploited extensively in the shipbuilding industry, it has not been used to any great extent in the oil industry. This may be because, as a process, it received most attention in Europe, while American engineers tended to concentrate on submerged arc, and since many of the early developments in constructional engineering for the oil companies were initiated in America, there has been a tendency to follow the welding practices of that country.

Gas Shielding

The search for a means of using bare wire without flux complications has led to the exploitation of inert gas shrouding. In open arc processes one of the major functions of any flux is to produce a large volume of gas, mainly CO_2, which protects the weld metal during transfer across the arc and while still molten in the weld pool. The slag formed by the flux continues the protection of the weld metal through the subsequent freezing and cooling period.

When dispensing with flux, a stream of inert gas can readily be used to replace the envelope shrouding the arc plasma, but the same protection cannot be applied to the solidifying weld metal as it passes outside the effective concentration of gas. Argon, which is entirely inert, is commonly used for aluminium, stainless steel, and specialized alloys, with either a consumable electrode or with filler wire and a tungsten arc. Argon also has the attraction that it promotes ionization and conductivity in the arc stream.

For mild steel fabrications, however, argon is too expensive, particularly when it is recognized that the very nature of the application demands that the whole arc zone must be wastefully flooded by the gas. The alternative is carbon dioxide, which is relatively inexpensive and is now extensively used in steel construction. As far as the wire is concerned, it is common and desirable practice to use a composition

which contains controlled quantities of silicon, manganese, or other de-oxidizers.

Bare wire CO_2 welding in mild steel is generally limited to light gauge structures, and the rather beady nature of the deposit militates against multi-pass welds. The quality of the weld furthermore depends entirely upon the preservation of the gas shield, and there are therefore hazards in using the process out of doors or in situations where the gas stream can be dispersed or deflected.

CO_2 *Welding with Fluxed Wire*

To expand the scope of CO_2 shielding fluxed wire can be used to advantage. The flux may be entrained in the interstices of a spiral winding surrounding the main core wire, as previously described, or be present as a core within the electrode. The latter method has the advantage of preserving the metallic outside surfaces for easy electrical contact and for feeding purposes. Fig 1 shows a section of such a wire formed from strip with the flux entrapped in the internal convolutions. A very important feature of this wire is that the folding and re-folding of the strip causes a distribution of the flux through the section which keeps the flux in intimate contact with the metal and aids the action of de-oxidizers and ionizers in the arc stream and weld pool.

The use of flux in addition to CO_2 shielding makes the weld quality much less sensitive to plate condition and gives consistent control of the bead contour. By virtue of the ionizers which can be introduced in the flux it also permits the use of an ac welding circuit.

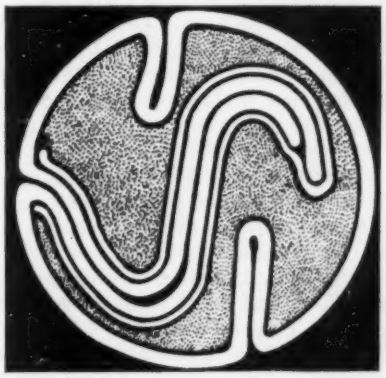

FIG 1

SECTION OF FLUX CORED WIRE AS USED WITH THE CO_2 SEMI-AUTOMATIC PROCESS

Because the flux does not have to generate large quantities of arc shrouding gas, it is relatively small in volume, and the composite wire can thus compete with bare wire for semi-automatic welding. The term semi-automatic is used in the sense that the wire is automatically fed into the weld pool, but the nozzle, usually in the form of a pistol, is supported and directed manually.

Electro-slag Welding

The methods of automatic welding so far discussed are primarily designed for operation with the work in the flat position and the direction of progress in a horizontal plane. As will be described later, they can be extended to the horizontal/vertical position

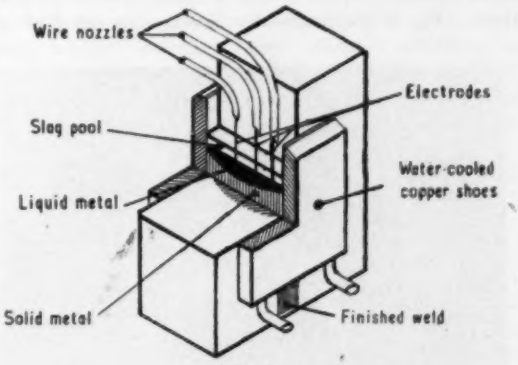

FIG 2

DIAGRAM OF ELECTRO-SLAG PROCESS

by suitable adaptation, but in most cases true vertical welding is not feasible. For this reason recent efforts have been directed towards techniques offering solutions to the problems of vertical welding. The successful processes in this field have been developed from the use of water-cooled shoes to contain the weld metal. One of these, the electro-slag process, was conceived originally as a means of welding very thick plates and forgings, and the fact that the material had to be set up vertically was dictated by gravity rather than design. Nevertheless, the electro-slag machine achieved the first truly vertical automatic welding, although its field of usefulness is limited.

The principle involved is shown in Fig 2. Here the heat is generated not by means of an arc but by the resistance of the molten slag. The plates to be welded are set in a vertical plane with square edge preparation and a separation of about 1¼ inches. The water-cooled shoes are clamped across the gap and are mechanized to travel upwards as welding proceeds. Welding is initiated by creating a pool of molten weld metal by a direct arc under a layer of flux. When sufficient head of molten slag is created the arc gives way to fusion by resistance, that is I^2R energy. Penetration is controlled by the electrical energy put into the melt, but the consistency of the penetration across the section of the material is regulated by imparting to the wire an oscillatory movement between the shoes and a controlled delay at each reversal of direction. A single wire is used for

plate thicknesses up to about 4 inches, but for greater thicknesses, which can rise to 14 or 15 inches, two or three wires in tandem are used, each supplied from a separate phase of a polyphase ac power source. Bare wire with the addition of granulated flux may be used, but the preferred method is to use a flux cored wire such as shown in Fig 2, usually $\frac{1}{32}$ inch dia. This gives greater scope for adjusting the properties of the weld metal, particularly in relation to the inherently large parent metal dilution and the tendency to low impact values expected from a weld of these proportions. Fig 3 illustrates the electro-slag machine at

FIG 3

VIEW OF ROCKWELD-VUS ELECTRO-SLAG MACHINE

work and shows one of the shoes and wire guide. The potentialities of the process may well revolutionize the design of large pressure vessels and heavy forgings.

CO_2 *Vertical Welding*

Another process of vertical welding uses the general mechanism of the electro-slag machine, and indeed was developed therefrom, but the fusion is by open arc in a carbon dioxide atmosphere. The principle is shown in Fig 4 which also illustrates the method of introducing the CO_2 gas. A synthetic wire of the type shown in Fig 2 is used, but it contains only a very light flux comprising de-oxidants only and forming little slag. This process uses a dc arc, not the least important reason being the problem of coping with the excess slag which would be formed by the

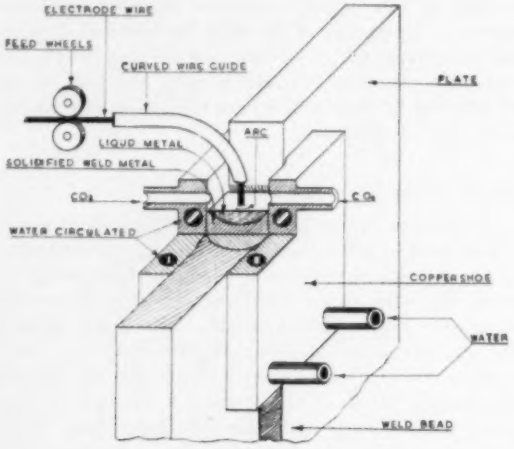

FIG 4

DIAGRAM OF CO_2 VERTICAL WELDING PROCESS

ionizers necessary in ac fluxing. It reduces the complications of electro-slag welding and, more important to the oil industry, it brings the scope of automatic vertical welding down to more practical plate thicknesses. It can in fact be applied to plates $\frac{1}{2}$ inch thick and upwards.

Machine Characteristics

For the fabrication of steel structures the preceding paragraphs outline the basic methods which welding engineers have at their disposal. Use is made of both ac and dc welding circuits, with their differing characteristics, and the range of practical welding currents may lie between 120 and 2000 amp, with welding voltages from 20 to 45 V. For manual welding a current limiting drooping volt/amp characteristic is essential, and to give stability and maximum aid to the welder it should be relatively steep, with open circuit voltage in the region of 65–75 for dc and 80–90 for ac. A similar characteristic is used for submerged arc and fluxed wires but rather flatter. In these cases a fixed arc voltage is selected and the current is stabilized by the automatic adjustment of the rate of wire feed to maintain the selected voltage. For special cases a flat or slightly rising volt/amp characteristic is used which becomes self-stabilizing when associated with a fixed but pre-set wire feed speed. It is a practical device for thin bare wire CO_2 welding, and is also applied conditionally to electro-slag.

In these various systems control of weld shape, penetration, and other dimensional features is achieved by selection of wire diameter, current, welding voltage,

and traverse speed. Fig 5 shows typical effects of these variables on submerged arc butt welds. Fig 6 shows comparative weld metal deposition rates for

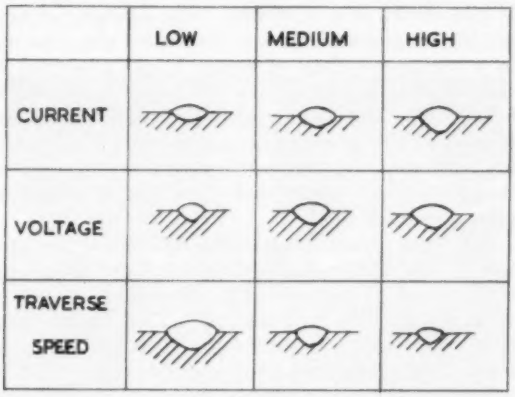

FIG 5

EFFECT OF VARIABLES ON WELD CONTOUR WITH SUBMERGED ARC PROCESS

various processes and demonstrates the productivity potentialities of automatic welding in the present known forms.

PIPELINES

"Stove Pipe" Welding

The need to construct pipelines to carry crude oil over enormous distances in remote and difficult terrain inevitably stimulated the development of techniques which would do the job in the most economical and expeditious manner possible. These circumstances have so far defied the application of mechanized welding, but they have produced the so-called "stove pipe" method of manually welding circumferential seams. The electrode used has a relatively thin and highly cellulosic coating and is deposited by dc arc at high welding currents. The procedure is to weld continuously from the twelve o'clock to the six o'clock positions in both directions. The characteristics of the flux enable a close deeply-penetrating arc to be held which with correct edge preparation permits the all-position working and gives an acceptably smooth internal bead. Careful selection of welders and intensive training is also an essential, since more than a little skill is required to make correct use of the properties of this type of electrode.

Mechanized Pipe Welding

There have been attempts from time to time to mechanize site welding of pipes, but they have not been very successful. The tolerances on diameter, thickness, and ovality of commercially available pipes makes it difficult to mechanize the process, quite apart from the intrinsic difficulties of positional operation and of controlling underbead shape in any mechanical welding operation. It would therefore appear unlikely that automatic welding as known at present will displace "stove pipe" technique for pipe welding, particularly when to the natural difficulties are added those of devising equipment for high-speed mobility on remote and often unkind terrain.

But there is always the possibility, particularly in the oil industry, of a fundamentally new process being

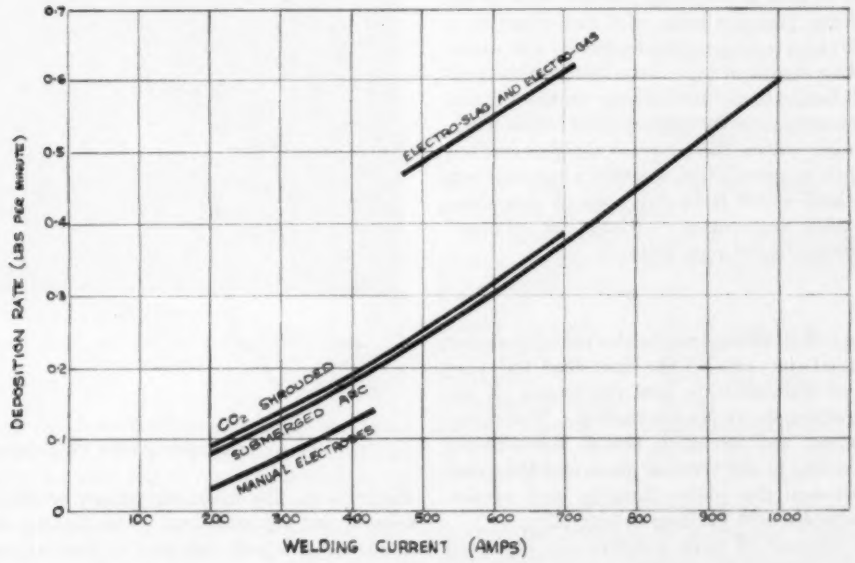

FIG 6

COMPARATIVE DEPOSITION RATES OF VARIOUS WELDING PROCESSES

developed permitting the operation to be carried out mechanically. There is, for instance, and as yet untried in the U.K., the new rotating arc process for circular welding involving an arc between butting pipe ends motivated by a rotating magnetic field and an upsetting action applied on the attainment of correct

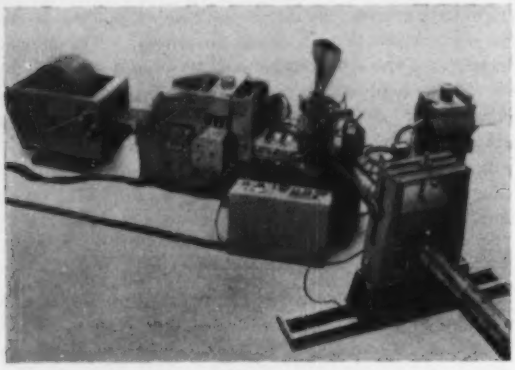

FIG 7
VIEW OF DRIAM CONTINUOUS SPIRAL TUBE WELDING MACHINE

temperature. While problems of applying this theory remain substantially unsolved, its future can only be one of speculation.

Another alternative to conventional pipeline laying is opened up by the method of continuous pipe fabrication from strip. The machine shown in Fig 7 forms strip into a close spiral which is welded by the submerged arc process. Two welding heads are used, one arranged so that it forms an internal weld as the strip leaves the forming rolls, and the other in a manner such as to complete the fusion by our external weld. As a means of pipe manufacture this technique is well established, particularly on the Continent, but has not apparently been applied to site work. Oil pipelines are within the scope of the process, but again it is open to speculation whether a machine can be devised which would have any hope of competing in speed, mobility, and economy with the "stove pipe" welding of prefabricated pipe sections.

Girth Seams

The welding of oil storage tanks also remains almost entirely manual, in spite of the fact that the very proportions of the structure and the length of the seams invite attempts at mechanization. The deterrents, of course, are the girth seams necessitating horizontal welding in the vertical plane and the vertical joints between the plates forming each strake. These problems are now yielding to ingenuity.

A certain amount of tank construction has been successfully carried out by a girth seam welder using the submerged arc process with the wire in the so-called three o'clock position. This machine consists of two welding heads, one inside and one outside the tank, mounted in a frame which straddles the tank wall and which is propelled along the upper edge of each strake as it is welded. The difficulty of supporting the granular flux and the molten slag is solved by a continuous chain of copper blocks presented to the tank wall in a manner which forms a substantial ledge. This machine has proved economically advantageous on the larger tanks, particularly on the multi-pass runs required for the thicker plates. It has also brought sounder welds and a marked reduction in plate distortion.

The main disadvantage of the submerged arc process for this application is the difficulty of setting up such a large weldment with sufficient accuracy to allow the arc to remain centred in the edge preparation. The additional difficulty of conserving and feeding the granulated flux under exposed weather conditions is not insuperable, but must be regarded as somewhat of a hazard to perfection.

The advent of CO_2 welding in association with flux cored wire such as shown in Fig 1 has permitted a step forward in this difficult positional work, and the latest girth seam welder, which is illustrated in Fig 8, exploits it. By using a visible arc and welding heads capable of manual adjustment while the arc is under

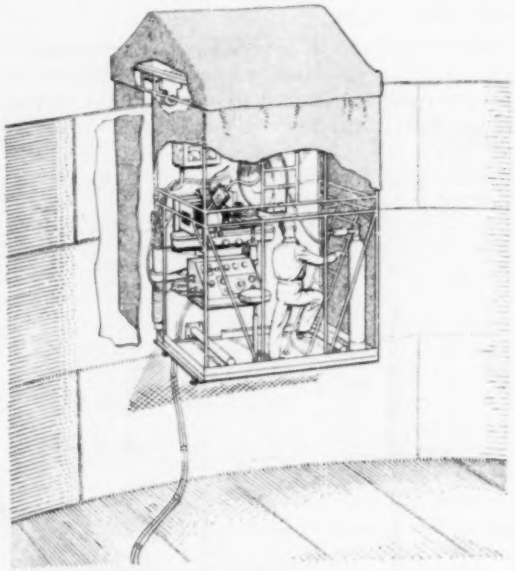

FIG 8
VIEW OF CIRCOMATIC GIRTH SEAM WELDER

observation, the main objections to the blind submerged arc are removed. The fluxing is not voluminous, but is just sufficient to form an encasing slag which, with the copper chain ledge, supports the weld metal as it freezes. This is shown diagrammatically in

Fig 9, while Fig 10 shows the caterpillar type copper chain forming the supporting ledge. The wire brushes, also shown in the latter illustration, are used to break the effect of wind and air currents tending to disperse the CO_2 gas.

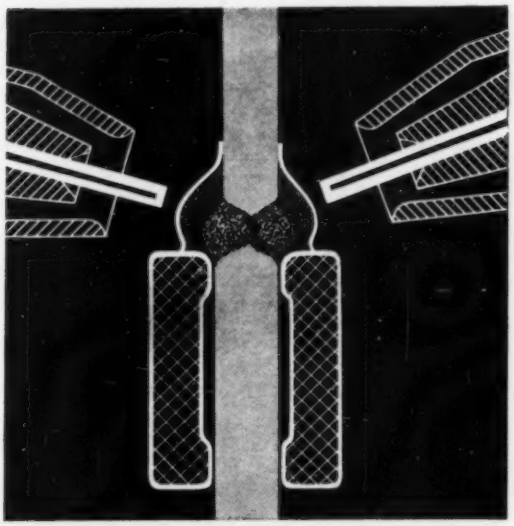

FIG 9
DIAGRAMMATIC SECTION OF GIRTH WELD

FIG 10
SLAG CHAIN AND ARC BOX OF CIRCOMATIC GIRTH SEAM WELDER

The welding heads are positioned so that one leads the other by about 15 inches, which means that the penetration on the root run is under observation by the operator of the trailing arc from the reverse side. This permits corrections to be made as welding proceeds to ensure complete fusion of the two roots. Independent controls in each cab and telephonic communication between the operators introduce the supervisory human element, which is still a vital factor in the art of welding.

Fig 11 tabulates welding data for girth seams of typical storage tanks. It is useful to note that the power and penetration of the CO_2 process, even in the three o'clock position, enable plate thicknesses of $\frac{3}{4}$–$\frac{7}{8}$ inch to be welded by single pass. It is also useful to record that, although outside the normal scope of storage tanks, this girth seam welder has been used on

PREPARATION	A	B	C	PASS Nº	WELDING AMPS OUTSIDE	WELDING AMPS INSIDE	SPEED INS/MIN	WIRE SIZE	WIRE LBS/FT OF SEAM
	1/4	1/4	–	1	380/420	420/450	24/30	1/8	0.24
	1/4	5/16	–	1	430/450	460/500	24/27	1/8	0.30
	5/16	3/8	–	1	450/470	500/520	24/27	5/32	0.32
	3/8	1/2	3/16	1	480/500	520/540	22/24	5/32	0.36
	1/2	5/8	5/32	1	500/520	530/550	20/22	5/32	0.40
	5/8	3/4	5/32	1	580/600	600/620	16/18	5/32	0.57
	3/4	7/8	1/8	1	600/650	620/670	12/16	5/32	0.77
	7/8	1	3/32	1	450/500	550/600	24/28	5/32	0.65
				2	450/500	450/500	24/28	5/32	
				3	400/500	430/530	26/30	1/8	0.29
	1	1 1/8	1/16	1	450/500	550/600	14/18	5/32	1.23
				2	500/600	500/600	12/16	5/32	
				3	400/450	400/450	16/20	1/8	0.72
				4	350/450	350/450	20/27	1/8	
	1 1/4	1 5/8	1/16	1	450/550	450/550	14/18	5/32	1.98
				2	500/600	500/600	14/18	5/32	
				3	450/550	450/550	16/20	5/32	
				4	400/500	400/500	18/24	5/32	
				5	350/450	350/450	20/26	1/8	0.56
				6	350/450	350/450	24/30	1/8	

FIG 11
PROCEDURE AND PERFORMANCE TABLE FOR GIRTH SEAM WELDER

vessels, such as blast furnace shells, welding wall thicknesses of $2\frac{1}{2}$ inches.

The mechanical properties obtained with the CO_2 process and the flux cored wire specially designed for three o'clock welding are:

Tensile strength	30–34 tons/sq in
Yield point	20–24 ,, ,,
Elongation	30–35 per cent
Reduction of area	50–65 ,, ,,
Charpy V-notch	45–70 ft lb at 10°–20° C.

Vertical Seams

The success with automatic girth seam welding has revived the question of automatic vertical welding. The CO_2 vertical welding process mentioned earlier has only just emerged from the development stage, but it would appear to promise successful service on

tank construction. In its present stage of development the welding of plates less than $\frac{1}{2}$ inch thick is not really practical, but this limitation allows ample freedom for taking over costly and laborious multi-pass welds on the lower strakes of large tanks.

Fig 12 shows the layout of the machine with its control panel, and Fig 13 shows a suggested method of setting it up for tank welding. Unprepared square-cut plate edges are used, and the plates are clamped with a gap of about $\frac{5}{8}$ inch between them, irrespective of plate thickness. Some attention must be given to cleaning the faces of the plates on which the water-cooled shoes run and, if the plates are stepped with respect to the lower strake, special preparation must be made to start the weld. Similarly, short run-off tongues, which are subsequently cut off, must be provided at the top.

For plate thicknesses less than 1 inch the wire is centred in the space walled by the two plate edges and

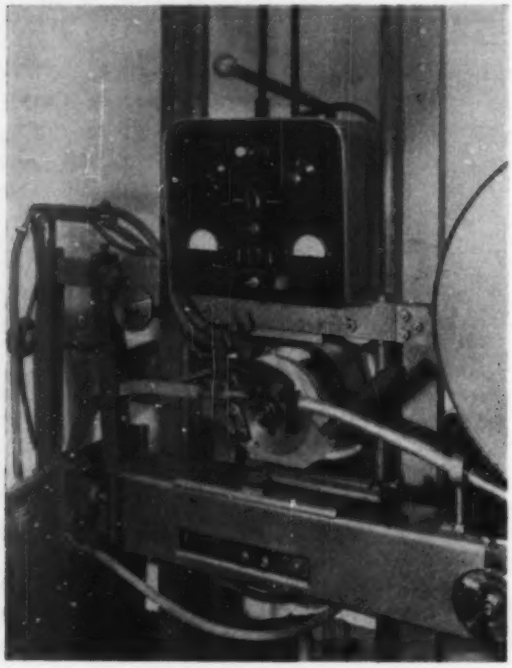

FIG 12

VIEW OF ROCKWELD VERTOMAT CO_2 WELDER

the two shoes. When the thickness is greater than 1 inch an automatic device for oscillating the wire and timing the delay at each reversal point is brought into use.

Apart from the advantage of speed when compared with manual welding, this automatic process offers the attractions of a single-pass weld irrespective of thickness. It ensures complete absence of slag inclusions, it gives even and fully controlled penetration, and it gives almost complete freedom from distortion.

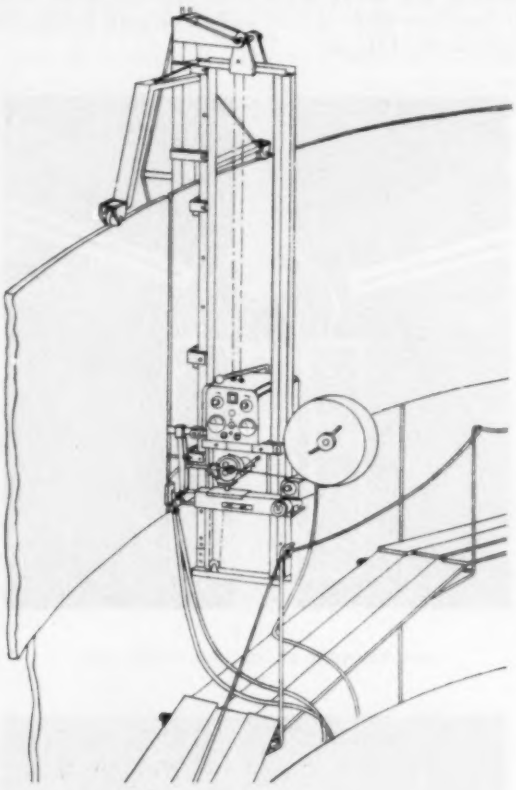

FIG 13

SUGGESTED MOBILE FRAME FOR APPLICATION OF VERTOMAT TO VERTICAL SEAMS ON TANKS

Using the flux cored wire designed for the purpose, the following mechanical properties are obtained:

Tensile strength	31–34 tons/sq in
Yield point	22–26 ,, ,,
Elongation	30–35 per cent
Reduction in area	50–65 ,, ,,
Charpy V-notch	40–70 ft lb at 20° C

TABLE II

Procedure and Performance Table for Vertomat Vertical Welder

Plate thickness, inches	Current, amp	Voltage	Gap between plates, inches	Welding speed, ft/hr	Deposition rate, lb/hr	Amplitude of oscillation, inches
$\frac{1}{2}$	400	27/30	$\frac{3}{4}$	18·5	17·6	
$\frac{3}{4}$	460	28/31	$\frac{3}{4}$	17·5	20·1	
$\frac{7}{8}$	550	29/32	$\frac{3}{4}$	16·5	25·2	
1	580	30/33	$\frac{3}{4}$	14·3	27·0	
$1\frac{1}{4}$	600	30/34	$\frac{3}{4}$	12·4	33·0	
$1\frac{1}{2}$	650	31/35	$\frac{3}{4}$	11·8	33·0	$\frac{1}{2}$

Table II tabulates the general performance data for this method of welding.

Tank Floors and Roofs

The flooring and covering of tanks do not invite mechanization to quite the same extent as the other weldments that have been mentioned. The construction adopted usually entails relatively thin lapped plates so that the welds are fillets with $\frac{1}{4}$ or $\frac{5}{16}$ inch leg lengths. The pattern of plate edges does not lend itself to economic setting up for self-traversing automatic heads, but the nature of the fillets does allow high deposition rates in manual welding, particularly if the newer iron powder electrodes are used. The overall economy of manual welding is likely to be unassailable until a new approach is made to the flexibility of the automatic processes or unless tank designers find cause for using very much thicker plates for floors and covers.

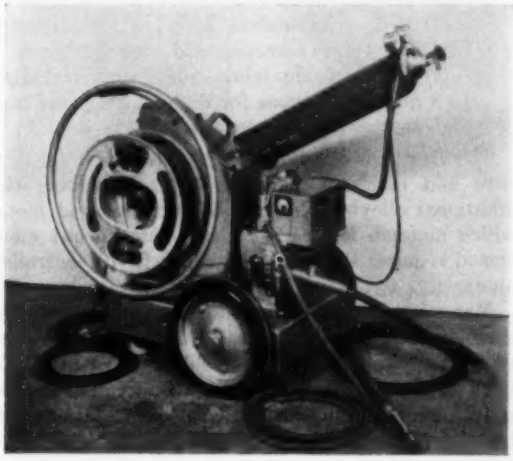

FIG 14
TYPICAL CO_2 SEMI-AUTOMATIC WELDING EQUIPMENT

There is, however, a compromise to be considered in the shape of the semi-automatic plant which may well offer sufficient speed and flexibility to reduce costs. In such an application, the flux cored wire with CO_2 shroud promises the best results. It is essential to employ a process which can be used out of doors and which can also produce sound welds on plate which has suffered exposure to inclement atmosphere. This in itself demands a de-oxidizing flux and a protective slag such as is afforded by the wire shown in Fig 1. Consistently sound welds of low hydrogen calibre are obtained with mechanical properties similar to those of the girth seam welding previously mentioned.

Typical equipment is shown in Fig 14. It consists of a wire feeding unit of the type which is self-regulating with respect to arc voltage, mounted on a mobile carriage with wire spool and CO_2 bottle. The unit shown is a universal machine which may be used with any standard ac or dc drooping characteristic welder of suitable capacity. From the wire feed rolls in the head the wire passes through a flexible hose to emerge concentrically through a CO_2 gas nozzle, to which the operator's hand grip is attached.

The wire used is $\frac{1}{8}$ inch od, which gives adequate flexibility in the hose, which for both mechanical and electrical reasons is restricted to about 20 ft in length. Bottled CO_2 supplied commercially cannot be free from a certain amount of water vapour, and to prevent porosity in the weld from this source it is the custom to install a small electric pre-heater in the pipeline, usually adjacent to the gas regulator.

Effective welding currents are in the 450–550-amp range. At these currents, welding traverse speeds are too high for the control of light butts and fillets in very thin plates, and hence semi-automatic welding in this particular form must be regarded as suitable only for $\frac{5}{16}$-inch thick plate upwards, a limitation which does not intrude upon storage tank practice. Table III gives data on performance.

TABLE III
Deposition Data
of CO_2 Semi-Automatic Welding Equipment

Plate thickness, inch	Preparation	Welding current, amp	Arcing time/ft of joint, min	No. of runs each side	Av speed/pass, in/min
$\frac{5}{16}$	fillet	500	0·43	1	28·0
$\frac{1}{4}$	fillet	,,	0·67	1	18·0
$\frac{5}{16}$	fillet	,,	1·00	1	12·0
$\frac{1}{4}$	Square close butt	,,	1·1	1	22·0
$\frac{5}{16}$	,, ,,	,,	1·3	1	18·5
$\frac{3}{8}$	Square $\frac{1}{16}$-inch gap	,,	1·7	1	14·5
$\frac{1}{2}$	60° double vee $\frac{1}{4}$-inch nose	,,	2·75	2	17·0
$\frac{3}{4}$	,, ,,	,,	4·15	3	17·0
1	,, ,,	,,	6·35	4	15·0

REFINERY AND CHEMICAL EQUIPMENT

Pressure Vessels

The majority of drums and pressure vessels used are of a type and size which can be constructed entirely in the contractor's workshop, and the main welding consists of circumferential and straight-longitudinal seams. The general approach to mechanization of the welding of such vessels is shown in Fig 15. As plate edge preparation and "fit-up" can usually be done with accuracy, the submerged arc can conveniently be applied, but there is no reason against an open arc with fluxed wire or CO_2 shrouding. With modern fluxes and wires sound radiographic welds present no difficulties with single or multi-pass welds in mild and low alloy steels.

Fig 15 shows a welding head mounted on a retract-

able boom, which in turn can be positioned vertically on a column. Longitudinal seams inside and outside are performed by the movement of the boom while the vessel is stationary. For circumferential seams, again inside or outside, the vessel is rotated on power-driven rolls while the welding head is stationary. For medium vessels of up to $\frac{3}{8}$ inch thick, square edge

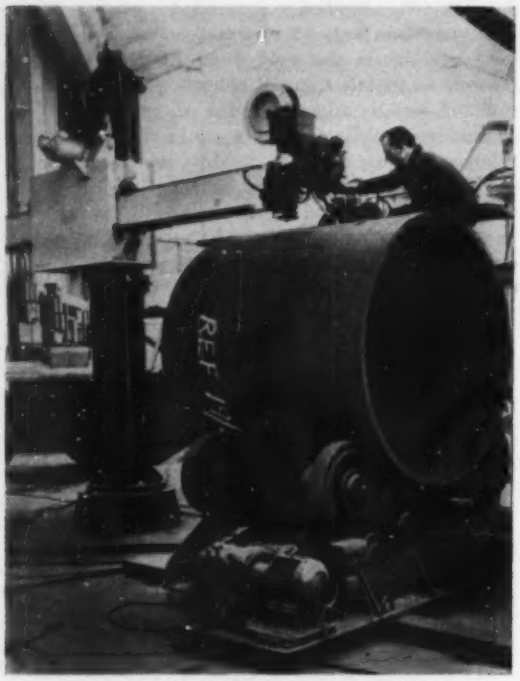

FIG 15
SUBMERGED ARC WELDING OF CYLINDRICAL VESSELS

plates can be used, but above that size edge preparation is necessary. Over $\frac{1}{2}$ inch thick, multi-pass welding is used.

In recent times experimental work has been done in the field of welding thick wall vessels, $2\frac{1}{2}$ inches upwards. This has been done both laterally and circumferentially by the electro-slag process. In the former case the rolled plates or forgings are set up vertically and the electro-slag shoes are curved to the contour of the vessel. Vessels of 30 inches inside diameter and 12 ft long with 3-inch wall thickness have been made in this way. In the second case, circumferential welds have been made with the electrode wire applied more or less tangentially to the gap so that the molten pool is at the three o'clock position and the wire fed into it vertically. Special shoes and starting and finishing devices are employed, while the vessel itself is rotated. This technique can be applied to both small and large diameter vessels with wall thicknesses from 2 to 12 inches.

Alloy Steels and "Cladding"

Many of the processes encountered in refineries demand special steels for reasons associated with high temperature or corrosive fluids. For these purposes, the greater use of "clad" steels, comprising a skin of the special alloy on a substantial mild steel backing, have been a feature of petrochemical developments and have brought their own problems to welding.

These special steels can be classified in general terms as:

(*a*) Low alloy chromium–molybdenum for creep-resisting properties.

(*b*) Medium chromium with low molybdenum for intermediate corrosion and heat resistance.

(*c*) Austenitic chromium–nickel steels including 18/8 and 25/20 types for difficult corrosion and high temperature conditions.

The first two can be welded with mild steel core wire and carefully balanced coating carrying the additional alloying elements to produce weld metal which matches the parent plate, but the last mentioned requires stainless steel core wire of controlled analysis.

Manual welding predominates in the fabrication of vessels, pipe systems, and heat exchangers using these materials, often because the weldment does not lend itself to a mechanized process, but also because the problems of securing the correct weld metal analysis by automatic means have yielded but slowly to solution. Much progress, even if fairly recent, has been made in this connexion, and both submerged arc and flux cored wire processes can be applied when need arises.

In the particular case of the "clad" steels it is essential to avoid dilution of the alloy weld metal by the mild steel backing, and for that reason the preferred practice is to use manual welding on the clad side in association with a special preparation and using light gauge electrodes for root runs. Root runs on the mild steel side are similarly applied by hand, but subsequent building up is readily done by any of the automatic processes if production demand merits it.

Root Welding of High Duty Pipes

Probably one of the most original yet simple contributions made by the welding industry to the art of welding pipes is the insert method of securing the correct root run. In many refinery applications it is essential that the bore of the pipe should be maintained smooth and free from irregular protuberances or crevices which by the nature of the fluids could initiate corrosion.

The insert method, developed originally for nuclear power installations, is illustrated in Fig 16, which shows (*a*) a section of the preparation and the insert

ring, and (*b*) a section of the finished root. The composition of the insert is selected to suit the pipe specification and a ring is formed of the pre-machined section. A tungsten arc is applied by manual torch and under an argon shield from the outside. Removable baffles are placed inside the pipe on either side of the weld, and the space within is purged by a further flow of argon. The ring is fused into the pipe and preparation without the addition of further weld metal. The action of fusion draws the bulbous section of the insert into the toes of the edge preparation of the parent

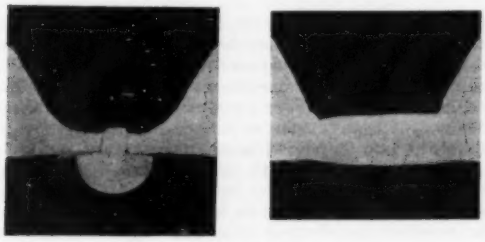

FIG 16
THE E.B. WELD INSERT

metal, and this physical condition is little affected by gravity. A consistent section is thus achieved for the full 360°. The actual bead contour inside the pipe is under the control of the operator, the variables being welding current and speed of traverse. An increase in current or a reduction in rate of progress will increase the edge fusion and draw the insert to a flatter contour, and hence both experience and judgment play a part in the correct use of this technique.

Once the insert root has been completed, the filling up and completion of the weld follows normal procedure.

CONCLUSION

Attention has been given in this paper to machines and techniques which are, as yet, not fully accepted in practice and, for that reason, they must be regarded as subject to the improvements which will inevitably follow their wider use. The electro-slag process, for instance, has been discussed in welding circles for some time, but at the time of writing and to the best of the authors' knowledge there is only one machine in the U.K. fully engaged upon a job of production. A number of other machines are commissioned and applied in experimental approaches to production problems, but are not yet in the hands of the shop floor personnel. Similarly, the only CO_2 girth seam welder in the U.K. has only just been pioneered through its first practical field job by one of the leading petroleum equipment contractors. Furthermore, the new vertical CO_2 machine, although established as a practical and economic proposition, still awaits the patronage of industry.

Progress in the welding sciences has, to some extent, moved more rapidly than the users' scope for assimilation and, consequently, there has been some understandable reluctance on the part of responsible engineers to become committed to ideas which may be obsolete before rightly put into practice. It is undoubtedly the task of industry to discriminate between the production aids offered, but there is also a responsibility to provide proving grounds. The present tendency to hold back in the hope or expectancy of something newer or better is not so pronounced on the Continent or in the U.S.A. and this has led to British engineers looking abroad for operating experience. In turn, this has led to the current general criticism that the U.K. lags behind in the race for new developments.

In summing up it is recognized that there are very many applications to which the only logical and economic approach must remain with the manual electrode. In fact, as a broad generalization and ignoring the potentialities of the new, very fine wire semi-automatic processes, it can be said that for any seam which can be completed in a single pass and on which currents in excess of about 300 amp cannot be used, mechanized welding is not likely to offer any production advantage.

The most profitable field for semi-automatic welding would seem to be in those weldments which can benefit from higher rates of weld metal deposition but to which the flexibility of manual manipulation must still be brought. Such cases, single or multi-pass, will utilize the "burn off" rates which go with 400–500 amp of, for instance, the semi-automatic CO_2 machine, either in slow traverse, broad weave, large volume per foot run, or fast traverse run of long footage per unit volume of weld metal. This versatility cannot be achieved with the thick core manual electrodes which would otherwise be used at these elevated currents.

When fabrication involves long multi-pass seams and elaborate plate edge preparation, full mechanization must be brought into consideration with the object of reducing, to unity if possible, the number of passes. The merits of a machine for the purpose must be judged primarily in the light of such aim, as this is the direct measure of the laborious man-hours which can be eliminated with the right equipment.

Little can be added to the broad descriptions given of various machines and processes without, on the one hand, introducing detail which is out of context, or, on the other, entering the realms of conjecture on the future. It may not, however, fall too closely into the latter field if, as a final note, attention is drawn to the recurrent use of the synthetic flux cored wire, Fig 1. It has been mentioned in connexion with the CO_2 semi- and fully-automatic equipment, with the electro-slag process, and the new open arc vertical machine.

The evidence, in general, shows that, despite

purging gases and special wire composition, it is necessary, as a practical economy, to have means of introducing controlling constituents fulfilling a very large number of objects, widely divergent in different processes. It would be impossible to cover all the variations without the aid of powder metallurgy which is now so easily applied in the synthetic wire. The scope would be seriously limited if the powders had to be formed into an adhering paste applied externally to the wire or if they had to be introduced separately and indiscriminately to the arc zone. Hence the flux cored wire has become a natural medium, and there seems little doubt that its exploitation has materially aided the advances in machine welding and may play an even greater part in the future.

DISCUSSION

G. W. Benzie (Esso Petroleum Co. Ltd): As is evident from the author's paper, welding is fast becoming a complex subject. To the non-specialist there would seem to be an almost bewildering range of processes and techniques available.

Herein may lie a danger in that this complexity may help to defeat the very object of this development, if only because people cannot make up their minds which way to go. Welding education, as I see it, is rapidly growing to paramount importance, and on this score the electrode manufacturers, the Institute of Welding, and now Cranfield are doing their best to meet this demand. However, I feel there are two aspects which are tending to become overlooked, attention to which I am sure will pay dividends.

The oil industry, and here I mean the refiners, really does welcome the introduction of new processes and techniques. We hope that it will mean cheaper, better quality fabrications on an improved delivery. However, if we are to help in providing those all-important proving grounds, I feel refinery engineers must have, in addition to an understanding of the process, full confidence in the mechanical and metallurgical properties of the resultant welds, at service temperatures and pressures.

Most weld metal properties published relate almost exclusively to room temperature. I feel now that there is an increasing need for more test data on low, and, particularly, on high temperature properties to be published, not only for carbon steel but also on the low alloy and stainless steel weld metals.

One chooses the parent plate with considerable care and sometimes trepidation, and it seems only reasonable that we should know that the weld metal will be at least as good under service conditions.

Turning to the practical applications of semi- and fully-automatic processes, such equipment can be, and usually is, mechanically complex.

If conditions are not right, particularly where one is relying on a gas shield, a lot of bad welding can be obtained quickly.

If confidence in the use of such equipment is to be built up, it is essential that users of such equipment employ the right type of operator and also ensure that the limitations, if any, of such equipment are fully understood by the shop floor.

Operating experience, if obtained at the expense of delivery and/or quality, will not help to break down any conservatism that may currently exist.

In concluding, I feel the authors are right in being enthusiastic over the advent of CO_2 welding. I am a little sorry they have not expounded more on the fine wire process using dip transfer conditions. For field construction in particular I consider that this technique offers great possibilities. One of the major advantages of the CO_2 process as I see it is that it offers an open arc technique with little or no slag and fume problems, and it can also be used semi-automatically. However, control of weld metal in the absence of a slag using spray transfer conditions can be very difficult, and hence such processes have not been fully positional. The use of a dip transfer technique has enabled this problem to be largely evercome, employing, as it does, a relatively cold arc with only a small quick freezing weld pool.

It would be of interest to hear the authors' views on the possibilities of combining the very obvious metallurgical advantages of a flux cored wire with dip transfer conditions. This would open up the possibilities of positionally welding not only carbon steel but also the low alloy and stainless steels.

W. K. B. Marshall: I agree with Mr Benzie that fabricators nowadays are faced with a bewildering number of welding processes, and I think it is a fair question to ask—why all these processes? The answer is that welding at the moment is in a state of flux, and all of us are feeling our way towards the ideal process or a small number of ideal processes depending on the application. As manufacturers, we see a need for a process, and as a consequence develop one to meet it. Whether in fact the new process meets the need or serves any useful purpose depends very much on how the user exploits it, and it is only by a process of trial and error and "ordeal by fire," as it were, that we really know whether or not a particular process does meet the need of the fabricator. These processes will, I feel, eventually sort themselves out, and one or two of the more important ones will come forward as meeting most of our requirements. There will always be the odd application which will justify the unusual process, but these will be in the minority.

In regard to the mechanical properties of welds at low and high temperatures, it does not matter very much how the metal is put down, if it involves depositing a large amount at one time there is always difficulty in achieving good impact properties at low temperatures. This applies to submerged arc welding, electro-slag, and electro-gas welding. It seems to be the feature of the very large weld pool involved in these cases, which in fact is similar to a heavy casting. One can achieve some improvement by alloy additions, but the overriding fact is that slowly cooled, large weld pools have poor low temperature properties. The properties can be raised to an acceptable level by a normalizing operation, but it is not often that this can be applied in practice. We have not abandoned, however, the possibility of finding a metallurgical answer to this problem. We have certain clues and can obtain good results sometimes. It is a question of ensuring good results at all times.

As far as high temperature properties are concerned,

I do not think there is any problem here. It is merely a question of chemical composition—chromium and molybdenum content—and, as we say in the paper, we can deposit by automatic methods weld metal with chromium and molybdenum contents as consistent as that in the parent plate. The creep resisting properties are, moreover, at least equal to those of the plate material.

I agree that gas shielding under windy conditions is a problem. It is one which has been recognized in one of the items of equipment we discussed, namely, the Firth seam welder. In order to combat high winds we arranged to have a sort of screen of metal brushes around the welding point so that the wind cannot get at the arc and blow away the CO_2 protection.

Mr Benzie asks for comments on the possibility of using flux cored wire under dip transfer conditions. The flux containing wire was developed originally, as far as CO_2 welding is concerned, to enable one to use any sort of power source, *i.e.* existing generators and transformers, and not to require the rather special dc power sources and other attachments needed for dip transfer operation.

With a suitably formulated flux, dip transfer technique is not required in order to obtain smooth welding, but it is possible that further improvement would result from a combination of the two processes. There is the practical point, however, that the flux cored wire would be difficult and expensive to produce in the fine gauges required for dip transfer technique. This economic factor might easily outweigh any technical advantage.

V. I. Holmes: One point which Mr Benzie mentioned is the simple but significant question of the required skill of the operators using these new machines. I do not think anybody need be put off by that aspect, because there is a marked improvement in the general knowledge and ability of welders throughout the industry in the U.K. I notice, particularly in the petroleum industry, that the quality of labour employed in welding is now much higher than that hitherto available, and I have seen very complicated machines used effectively by skilled welders with very little additional training.

R. C. Thomson (G. A. Harvey & Co. (London) Ltd): I would like to state that there is such a process on the market which uses a magnetically flux coated wire with CO_2 shielding. This process, I believe, can be adjusted for either dip or spray transfer.

C. A. Burden (British Oxygen Co. Ltd): As a member of the company concerned, I would like to say that dip transfer technique can be used with the Unionarc process.

E. Thornton (British Petroleum Co. Ltd): In regard to the welding of thick sections, have the authors had any failures which they could detail and, if so, where do they occur? I wonder if the authors have to allow special factors of safety in the design of structures using such welds.

W. K. B. Marshall: I presume that Mr Thornton is referring to failures in electro-slag welding of thick plates. We ourselves have not experienced any such failures, but most of the service information on this type of welding comes from Russia, where there is some ten years of practical experience. This does not mean to say that there are not now quite a large number of electro-slag welded structures in service, both in the U.K. and on the Continent. The Russians have reported some cases, not of failures, but of practical difficulties in making the welds of cracks and of porosity, the latter being due in most cases to deficiencies in the plate material. Such troubles, of course, are rectified before the job has gone into service, and I do not know personally of any actual failure of an electro-slag weld.

R. C. Thomson: I would be interested to know what degree of success has been obtained with the circumferential welding of the electro-slag process.

V. I. Holmes: The electro-slag machine has been applied to the circumferential welds of vessels with the axis of rotation in a horizontal plane. The application, however, has been in the experimental shop, and to the best of my knowledge has not been applied commercially. Although not quite the same thing, electro-slag welding has been applied to the bulk welding of large diameter solid shafts. A typical case is that of a crankshaft, about 12 inches in diameter. Two square plates slightly larger than the shaft diameter are bored and fitted to the shaft ends to give a box formation at the joint. The copper shoes travel on the outside of the box and, in effect, a solid square weld is produced. It is subsequently machined down to the shaft diameter.

J. M. Dougary (Kuwait Oil Co. Ltd): I was very interested to hear of the application of automatic welding on vertical seams in storage tanks. I wonder if the authors would like to say how long this new technique has been in use, where it has been used, and on what capacity tanks?

W. K. B. Marshall: It is only very recently that automatic welding has been applied to the vertical seams in storage tanks. The machine itself is very new, and it requires a little courage for a contractor to commit himself to its use. There are several machines operating in Europe, and one of the large fabricators in the U.S.A. has put on order more than a dozen machines for the same purpose. There have been certain teething troubles, associated not so much with the weld as with the quality of the plate to be welded. Defects in the plate material become much more important with this process and, in particular, laminations of the plates have to be watched and rectified prior to welding.

W. Bentley (British Petroleum Co. Ltd): Still on the subject of the welding of vertical seams in tanks, could the authors tell us whether, when a vertical seam is welded in this way, a satisfactory condition at the T of the two welds is obtained, or is it found necessary, as is often the case, to cut out the T and re-weld it by hand? This is, perhaps, one of the most troublesome problems encountered to-day in welded steel tanks.

V. I. Holmes: I do not think this presents serious difficulty. It is necessary, of course, that the face of the T is dressed off to get a smooth starting surface for the copper shoes. As far as the machine is concerned, the arc can be started directly in the bottom of the T.

W. K. B. Marshall: One of the advantages of the electro-gas process, as opposed to the electro-slag process, is that sufficient heat is developed immediately on striking the arc to achieve full fusion of the side walls, and thus no run-on plate is required. On the other hand, it is still necessary to have run-off plates in the form of

small plates extending the gap upwards. This device allows the shoes to run off the top of the plates, and by so taking the finish of the weld beyond the plate, it ensures that any crater trouble is outside the length of the weld.

C. C. Bates (Corrosion & Welding Engineering Ltd): I have a little experience of the CO_2 welding process on tanks. A complete tank farm was built in the Middle East, using this process on all the horizontal seams, and it is rather interesting to record that the percentage of rejects found when using this process was in fact lower than with any other automatic welding process. Another fact which the authors did not mention was that there is a plate thickness limitation on using this equipment on tanks, not so much in the process itself as on the top tiers of the tank, where the welding machine is hung on the top plates. When one goes below $\frac{5}{16}$ inch the plate starts to buckle with the machine weight, and therefore the welding head is thrown out of alignment.

Mr Holmes did mention a 12-inch advance between the front welding head and the back one, but I think that is a little on the large side.

On the question of welding the vertical seams, I think Mr Bentley has made the very valid point that one invariably has to weld a vertical seam in advance of the horizontal seam, and therefore one is starting off by welding a vertical seam with nothing to build it on. Welding horizontal seams in advance of vertical seams always leads to trouble at the T-joint, and I think one of the answers to this problem is to put a false coupon plate underneath the bottom of the vertical seam, which can be withdrawn afterwards, and then chip out and re-weld the entire T-section later.

A problem which is continually cropping up and on which I would like the authors' opinions is that with CO_2 welding one is inclined to obtain a very narrow weld, a weld of narrow shape and section, so that when a bend test is carried out because one has a narrow weld there is little or no ductility in the weld zone and too much stress taking place in the parent metal. There is a tendency for cracking at the edge of the weld, at the fusion zone, and it is a difficult problem to try to assess whether in fact the weld is a good one or not.

There is an application for fine wire CO_2 welding in pipelines, and I think an oil company in New Jersey has done quite a few miles of it. However, I would like to take issue with the last speaker in that it has been proved that fine wire welding is only in fact faster than a good manual worker on small diameter pipes.

W. K. B. Marshall: Mr Bates has reminded us that the best practice is to weld the vertical seams first, and I agree that starting blocks are necessary to close the horizontal gap and to aid the change in thickness of successive tiers. The problem of bend tests raised by Mr Bates is really one that should not occur, and I do not think will arise as we gain more experience in testing CO_2 welds. A CO_2 weld shows very deep penetration, and this gives a narrow parallel-sided weld section. Moreover, since CO_2 welds tend to be stiffer than the average run of manual arc welds, little or no deformation occurs in the weld metal during the transverse bend test. As Mr Bates suggests, this throws all the deformation required to form the bend on to the metal at the side of the weld, and one is then not testing weld metal but plate material. I would suggest that the best way of testing the weld is to use a notch bend test to check for defects within the weld or, if one wishes to check the ductility of the weld metal, to use a longitudinal bend test which deforms the weld irrespective of the stiffness of the parent metal. It might be difficult to get inspectors to change their habits in this direction, but these two tests are much more revealing than the classical transverse bend test.

T. H. Webster (Shell Refining Co. Ltd): I would like to ask how far CO_2 welding has been introduced and accepted in the petroleum industry, especially with regard to pressure pipe butt welding?

W. K. B. Marshall: I personally doubt very much whether CO_2 welding is being used to any extent in the oil industry. It may be used by one or two sub-contractors doing work for the oil industry, but to my knowledge it is certainly not used seriously on site work. Intelligently applied, however, I am sure it offers great advantages to the industry.

G. W. Benzie: If I may come in here, we at Fawley are experimenting with the fine wire welding process. We have done positional fine wire welds, but are not certain yet on the properties. We have not drawn any conclusions yet, but it has advantages; we reckon it will speed up the welding considerably. With its rolled joint it is no problem, but it has to be fully positional as far as we are concerned.

W. K. B. Marshall: On that point experience shows that as far as rate of welding is concerned, in the vertical position there is little to choose between the fine wire process and manual welding with No. 6 swg electrodes. However, one cannot always use a six-gauge electrode vertically, in which case the advantage as regards speed lies with the fine wire process.

Another imponderable advantage of the fine wire process is its ability to bridge wide root gaps. In the fabrication of water turbine volutes, for instance, fit-up is extremely difficult, and gaps up to as much as $\frac{1}{16}$ inch are difficult to avoid. One manufacturer of such volutes finds it economical to use fine wire CO_2 for the root run to bridge these gaps and to fill up by manual welding.

C. A. Burden: In our experience with pipe welding we have found and, in fact, have had competition within our own company between stove pipe welders and men skilled in the art of using fine wires and CO_2, that on smaller diameter pipes fine wire welding is very much faster. On pipes of 18 inches in diameter the difference in welding speeds between fine wire welding and manual arc tends to even off. A typical welding time on 8-inch diameter $\frac{1}{8}$-inch wall pipe is of the order of 10 minutes with dip transfer fine wire CO_2 welding, which is quite fast.

In regard to tolerance to gaps, it is a really remarkable process. I have seen a $\frac{3}{16}$-inch gap in a 20 gauge lap weld bridged quite easily.

In regard to the use of it in the petroleum industry, a lot of people, as Mr Benzie has mentioned, are experimenting with the process. Equipments have been sold to pipe manufacturers, who have found it extremely useful for putting in root runs prior to automatic welding the finishing pass. One manufacturer who bought equipment in the U.K. is now using it in New Zealand for that application.

The President then called for a vote of thanks to the authors, which was accorded with acclamation.

ELECTRON MICROSCOPY OF LUBRICATING GREASES *

By G. V. VINOGRADOV † and V. V. SINITSYN †

INTRODUCTION

It is commonly known that greases are two and sometimes multi-phase dispersed systems. They are made by thickening mineral or synthetic oils with solids. The materials used as thickeners may be soaps, solid hydrocarbons, inorganic products, or organic compounds other than soaps. The most important soap greases are the calcium, sodium, lithium, and aluminium types, the metal being the soap cation. The thickener content in greases usually varies from 5 to 25 per cent.

Great progress has been made in the study of greases during the past 10 to 15 years. The use of rheological methods, thermal and X-ray structural analysis, polarization, dark ground and phase contrast microscopy, has expanded our knowledge of grease structure. However, it may be stated without exaggeration that only the electron microscope made it possible to solve finally the question of grease structure.

The dispersed phase of a grease is usually found to contain anisometric micro- and sub-microcrystallites (ribbon- or needle-shaped, tabular, etc.). However, silica aerogels and certain other thickeners consist of chains of spherical particles. The structure of the dispersed phase may be modified by the use of additives.

When soap- and hydrocarbon-base greases are obtained from melts, crystallization results in the formation of a monolithic pseudogel, a structural skeleton made up of inter-grown and inter-entangled crystallites. The structural skeleton penetrating the entire bulk of grease specimens obtained from melts is easily broken up by mechanical influences. When this structure is destroyed, by milling for example, the dispersed phase is in the form of large unbroken fragments of the original structure together with smaller fragments [45,69-70,72]. The larger structures can be studied with the optical microscope.

The primary structure of grease, *i.e.* the morphology of their skeletal structure and the crystallites involved, can only rarely be studied by optical means, because they are usually too small to be conveniently examined. In homogenized greases the average particle size of the dispersed phase varies, as a rule, between 0·1 and 10 microns. Only in hydrocarbon and certain Na-greases do they reach a size of 100 microns and more, and therefore big enough to study under the microscope. The particles of the dispersed phase may be very thin, not more than hundredths of a micron thick and sometimes, it seems, even thinner.[82]

In studying the structure of the primary particles of the dispersed phase (crystallites) the most effective results are obtained by X-ray structural analysis and electron micrography. It was shown recently [95] that the structure of polymolecular layers of higher fatty acid soaps could be studied by electron microscopy.

The electron microscope, though a most important instrument in studying the grease structure, does not substitute other methods. Only the use of the entire range of modern means of studying plastic dispersed systems, to which greases belong, enables investigation of their structure. To establish the rational bounds of electron microscopy in the study of greases an examination must be made of all published data. This will make it possible to outline the main tasks confronting electron microscopy in this field.

This review is a summary of the results contained in papers published from 1941 to December 1960 and takes into account all the present authors' investigations. It is the first complete review of papers on electron microscope investigation of greases.

HISTORICAL

The structural skeleton of soap greases, the most common type, consists of crystallites of higher fatty acid soaps. The first electron photomicrographs of soap crystallites were obtained in 1941 by Marton, McBain, and Vold,[1] and later by Tiessen[2] and Hattiangdi.[9] In the first, the studies were conducted on 4·6 per cent aqueous solutions of sodium laurate. A 400-mesh screen was lowered into the soap solution, then taken out and dried under vacuum. The specimen was examined under the electron microscope and a typical photomicrograph of laurate soap is shown in Fig 1. It will be evident that the ribbon-like Na-laurate crystallites are very similar to the crystallites of Na-soaps constituting the dispersed phase of greases. Attention is drawn to the fact that there are as yet no comparative investigations of the morphology and structure of soap crystallites formed in aqueous and hydrocarbon (non-polar) media, nor on the effect of polar additives on the way the soaps crystallize in non-polar media. Such studies would be of great importance to the entire field of greases.

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Interesting possibilities are offered by the electron microscope for the study of the structure of polymolecular film structures using soaps of heavy metals.[95] Thin sections of the film were cut in the direction of the soap molecule axis. The magnification was about $2{\cdot}4 \times 10^5$. An alternation of dark bands can be seen in the micrographs, corresponding to the metallic cation layers. The distribution of dislocations in the polymolecular soap film is visible.

The first electron microscope study of soap grease structure was made in 1946 by Ellis.[4] Studying the structure of greases based on hydrated Ca-soaps (cup grease), he showed that the dispersed phase consisted of Ca-soap fibres in the form of a twisted two-stranded string (Fig 2). The excellent electron photomicrographs obtained by Ellis were published in Burton and Kohl's monograph.[3] At the same time, Farrington and Birdsall[5,6] published the results of electron microscope studies of the structure of the main types of soap greases. They showed that all these greases (except the Al-type) contain ribbon- or needle-like soap crystallites from 0·2 to 50 microns long. The length/breadth ratio may vary between 10 : 1 and 200 : 1. The data they published concerning the shape and size of the dispersed-phase particles of various soap greases have been confirmed by many subsequent studies.[10,19,41,64,66,etc.]

METHODS OF PREPARING SPECIMENS FOR STUDY UNDER THE ELECTRON MICROSCOPE

Ellis, Farrington, and Birdsall used the smear and suspension techniques for preparing grease specimens for electron microscope study, and most investigations still use similar or slightly modified techniques. The main methods of preparing specimens for studying greases under the electron microscope are reviewed briefly below.

Suspension Technique (Dry Method)

This technique, because of its simplicity and the good results obtained, is the most widespread to date.[67] A small portion of the grease (one part) is shaken in petroleum ether (approximately 200 parts) or in some other highly volatile solvent (*iso*-octane, ethyl ether, carbon disulphide, etc.) in which the dispersed phase is insoluble, but which readily dissolves the dispersion medium. In some cases, it is good practice to wet the grease specimen, before making the suspension, with a few drops of a low viscosity oil.[5,6] A drop of the suspension, containing separate particles of the dispersed phase and fragments of the de-oiled structural skeleton of the grease in a suspended state, is applied to a conventional specimen carrier (200 mesh metallic screen coated with collodium, formvar, or any other suitable film). After evaporation of the solvent the specimen carrier is placed in the electron microscope. It is convenient to examine all specimens with an optical microscope before proceeding to the electron microscope.

A development of this method of suspension formation has been described by Vold.[82] In this method the system is suspended in benzene or in *n*-heptane. After repeated centrifuging, using a fresh solvent each time, an oil-less suspension results.

The suspension technique gives very good electron photomicrographs. An illustration is the electron photomicrograph of a Na-grease shown in Fig 3.[41]

Some investigators[13,29,32,56,etc.] suggest that the actual structure of grease specimens prepared by the suspension technique, and to a lesser degree by the smear technique (see below) may be distorted. Such possibilities are exaggerated. In papers by Brown *et al*[21,22] and Farrington[19] the structure of grease specimens prepared by the suspension and smear techniques is shown to be practically identical to that of specimens prepared by the smear method without additional treatment with solvents. The elution of the dispersion medium does, however, show the dispersed-phase structure of the greases much more distinctly. Vold[54] established that the size and shape of the soap fibres remain unchanged even if the suspension is stored for a week.

Investigations on pure soaps have been made on suspensions formed in various solvents, water, alcohol, etc.[1,9]

The Smear Technique

In this method the grease is smeared on to a thin plastic or carbon film supported on a specimen grid. The grease layer must be thin enough to be transparent to the electron beam. The oil phase of greases scatters the electron beam considerably, resulting in poor contrast and definition. For this reason the grease smear is normally washed in solvents prior to examination. The micrographs obtained by the smear technique are no less distinct than when the specimens are prepared by the suspension technique.

An interesting modification of this method is the so-called print technique suggested by Mottlau.[10] A thin layer of grease is spread on a glass slide. The filmed specimen carrier is pressed lightly against this grease layer. Then the grease "print" on a collodium or formvar film is treated with solvents to elute the dispersion medium, as described above. The print method has been employed successfully for studying changes in grease structure right in the working zones of mechanisms. For example, prints can be made of the runways, rollers, and balls of roller bearings.[21,28,52,77] An electron micrograph of grease structure obtained by the print technique is shown in Fig 4.[52]

Elution of the oil from a grease may affect grease structure to some extent. Barwell[48] has suggested

FIG 1

ELECTRON PHOTOMICROGRAPH OF SOAP FIBRES OF SODIUM LAURATE OBTAINED FROM AQUEOUS SOLUTION[1]

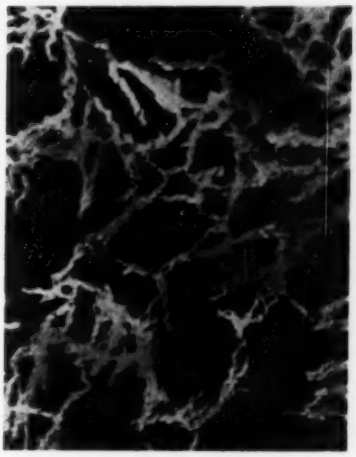


FIG 2

ELECTRON PHOTOMICROGRAPH OF A HYDRATED Ca-GREASE[4]

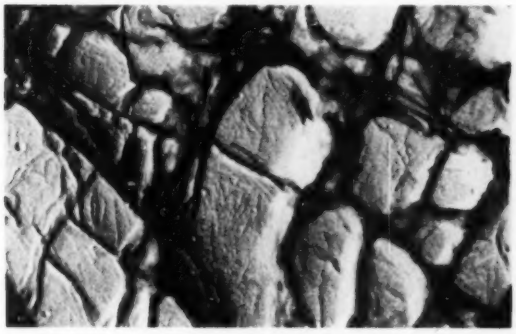


FIG 3

ELECTRON PHOTOMICROGRAPH OF A Na-GREASE. SPECIMEN PREPARED BY SUSPENSION METHOD

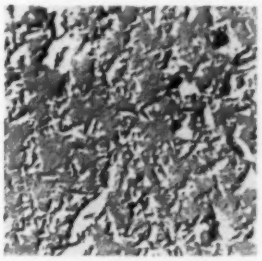


FIG 4

ELECTRON PHOTOMICROGRAPH OF A Li-GREASE SPECIMEN PREPARED BY PRINT METHOD DIRECTLY ON BALL-BEARING RUNWAY[52]

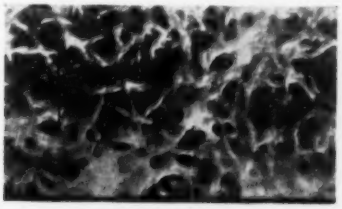


FIG 5

ELECTRON PHOTOMICROGRAPHY OF Li-GREASE. SPECIMEN PREPARED BY SLICE METHOD[38]

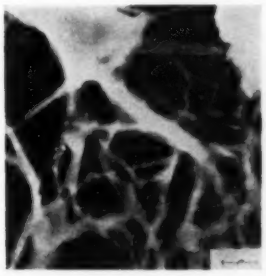


FIG 6

ELECTRON PHOTOMICROGRAPH OF Na-GREASE. SPECIMEN OBTAINED BY AEROGEL METHOD[55]

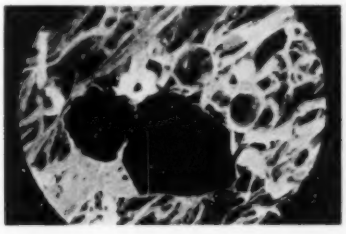


FIG 7

ELECTRON PHOTOMICROGRAPH OF HK-50 Na-GREASE. DARK GROUND IMAGE

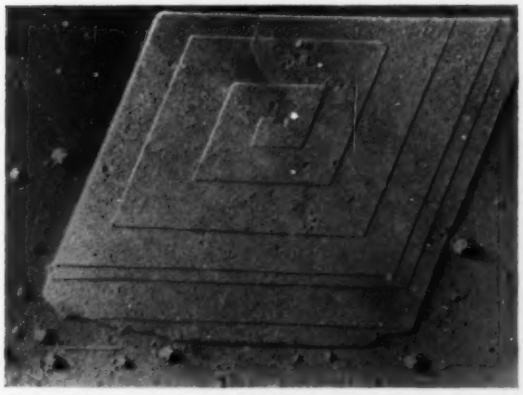


FIG 8

ELECTRON PHOTOMICROGRAPH OF THE STRUCTURE OF A CRYSTALLITE OF *n*-HEXATRIACONTANE $C_{36}H_{74}$[35]

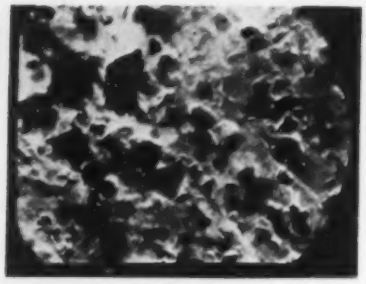


FIG 9

ELECTRON PHOTOMICROGRAPH OF BENTONITE GREASE

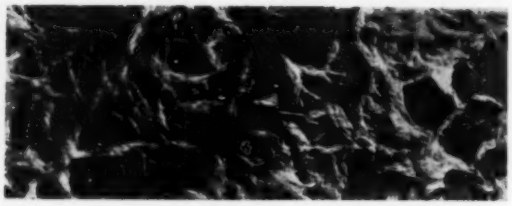
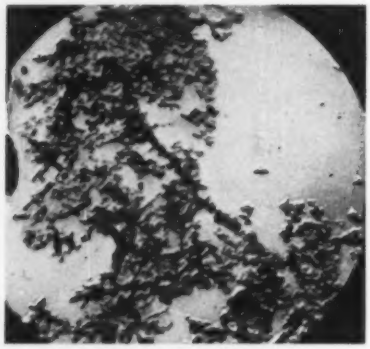
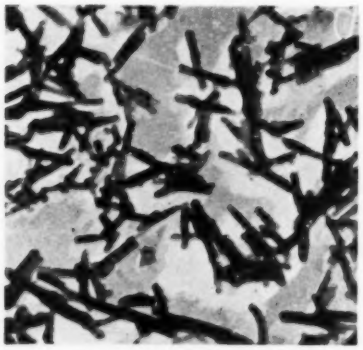


FIG 10

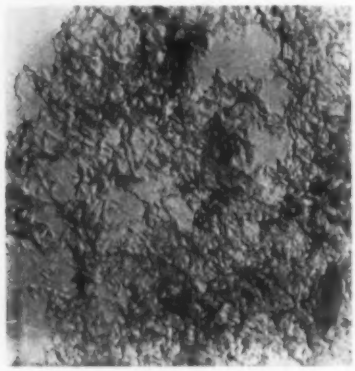
ELECTRON PHOTOMICROGRAPH OF A GREASE THICKENED WITH THE Na-SALT OF THE METHYL ESTER OF *n*-OCTADECYL-TEREPHTHALIC ACID[57]



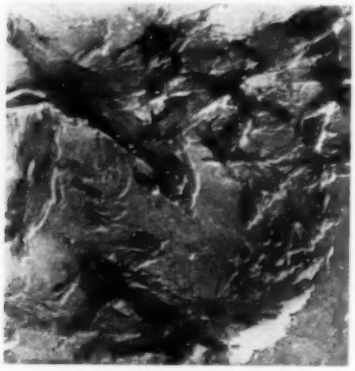
(*a*) Li-SOAP, 0·1 PER CENT NaOH



(*b*) Li-SOAP, NEUTRALIZED



(*c*) Na-SOAP, 0·1 PER CENT NaOH

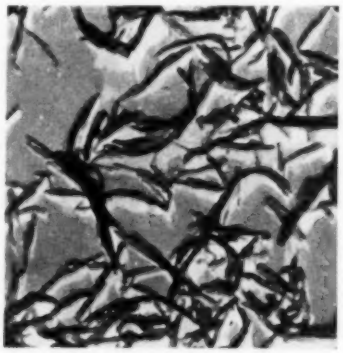


(*d*) Na-SOAP, NEUTRALIZED

FIG 11

EFFECT OF FREE ALKALI ADDITIONS ON THE STRUCTURE OF Na- AND Li-GREASES

(*a*) OIL OF VISCOSITY $\nu_{20^\circ} = 4{\cdot}1$ cst

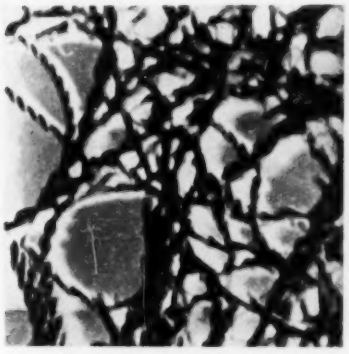


(*b*) OIL OF VISCOSITY $\nu_{20^\circ} = 1700$ cst

FIG 12

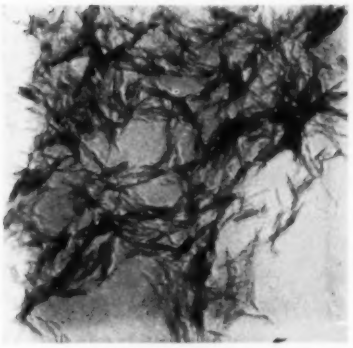
ELECTRON PHOTOMICROGRAPH OF Li-GREASE[69]

(*a*) Li-STEARATE



(*b*) Li-12-HYDROXYSTEARATE

FIG 13—EFFECT OF NATURE OF SOAP ANION ON STRUCTURE OF Li-GREASES

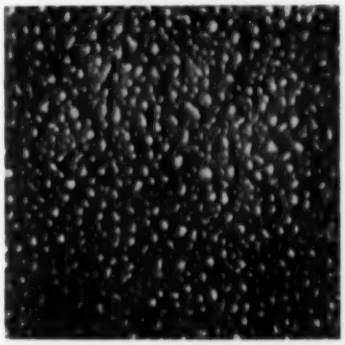


(*a*) RAPID COOLING

(*b*) SLOW COOLING

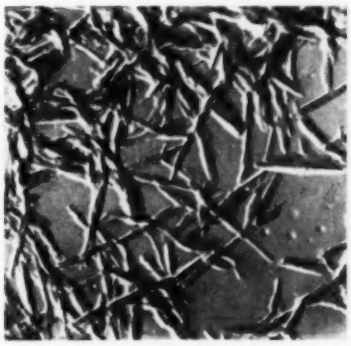
FIG 14—EFFECT OF COOLING RATE ON STRUCTURE OF Na-GREASES

(*a*) INITIAL GREASE



(*b*) GREASE AFTER BREAKDOWN AT DEFORMATION RATE OF $2{\cdot}5 \times 10^4$ sec^{-1}

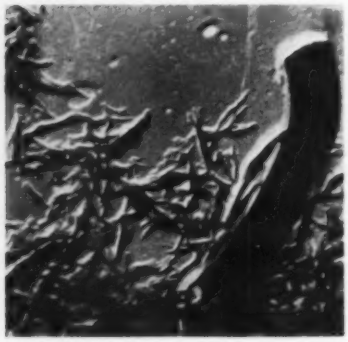
FIG 15—BREAKDOWN OF STRUCTURE OF CUP GREASE



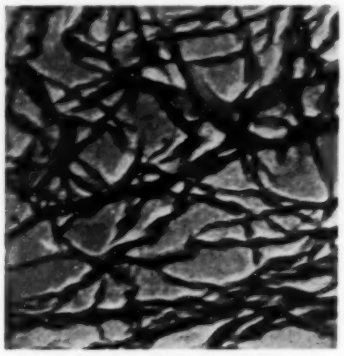
(*a*) INITIAL GREASE

(*b*) GREASE AFTER BREAKDOWN AT DEFORMATION RATE OF $2{\cdot}5 \times 10^4$ sec^{-1}

FIG 16—CHANGE IN Li-GREASE STRUCTURE DURING BREAKDOWN AND RESTING



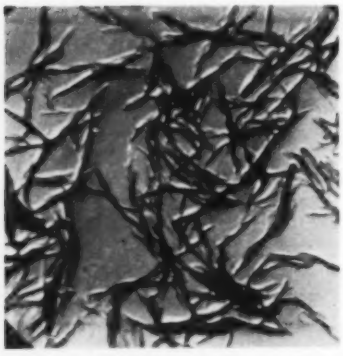
(*a*) 20°



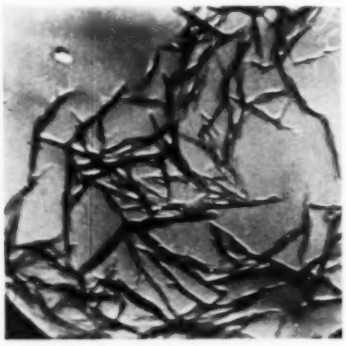
(*b*) 80°

FIG 17

EFFECT OF TEMPERATURE ON BREAKDOWN OF Na-GREASE STRUCTURE AT 3000 SEC^{-1}



(*a*) ONE MINUTE AFTER DESTRUCTION

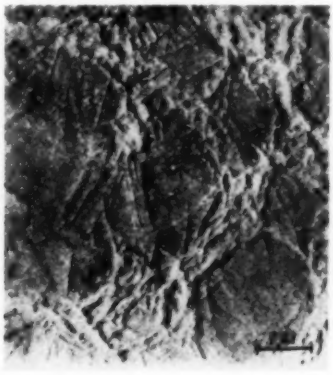


(*b*) ONE YEAR AFTER DESTRUCTION

FIG 18

EFFECT OF RESTING ON STRUCTURE OF Na-GREASE DESTROYED AT DEFORMATION RATE OF $2{\cdot}5 \times 10^4$ SEC^{-1}

(*a*) BEFORE IRRADIATION



(*b*) AFTER IRRADIATION ($7{\cdot}7 \times 10^8$ ROENTGENS)

FIG 19

DESTRUCTION OF STRUCTURAL SKELETON OF Na-GREASE BY γ RADIATION [60]

slow evaporation under vacuum for removing the oil from these grease prints.

Sectioning Technique

This technique has found wide use in the study of biological preparations. Sections for electron microscope investigations of greases must be very thin (about 0·1 micron), and it is difficult to prepare them from a soft lubricant. The method developed by Vold[13] for models of soap greases (cetane–Ca stearate system) consists in freezing the specimens at −70° C and obtaining sections 0·05 to 0·1 microns thick using a special microtome. The sections are floated on to specimen grids and examined under a microscope. The cetane is partly removed by evaporation under vacuum. The low quality of the image[13,20,26] has led to the suggestion in more recent papers[32,38] that the cetane could be more completely removed under vacuum or, alternatively, that benzene could be used to displace the cetane and the benzene subsequently removed by sublimation under vacuum.

The sectioning technique is not applicable to ordinary greases because petroleum oils do not crystallize like cetane even at temperatures of the order of −70° C. To study such systems, Vold has suggested[38] displacing the oil phase with cetane and then preparing sections as described above. An electron micrograph of a Li-grease obtained by the sectioning technique[38] is shown in Fig 5.

The low quality of the images produced by microtomy is the reason for most workers using other methods. The studies made by Vold *et al* with this technique are, however, of considerable fundamental interest, because they give a means of assessing other methods for freedom from distortion of the grease structure during specimen preparation. Contrary to the opinion expressed by Leet,[39] the micrographs of grease sections testify to the fact that the suspension and smear techniques do not distort the grease structure.

The sectioning method may evidently be used for studying secondary grease structures by optical microscopy.

Aerogel Technique (Critical Point Method)

By this method the dispersion medium can be removed completely from greases, leaving their structural skeleton intact as an aerogel. This is achieved by replacing the liquid dispersion medium by a gas. The method was first applied to greases by Browning,[12] who substituted highly volatile solvents (liquid butane or ethylene) for the oil phase at an elevated pressure in an autoclave. The grease specimen is then heated above the critical point of the solvent, which must be below the phase transition temperature of the dispersed phase. Under these conditions the solvent evaporates without forming menisci which would destroy the structural skeleton of the grease.

Peterson and Bondi[31] demonstrated that the structure observed with the electron microscope does not differ from that of the initial grease. It is now recognized that this technique can give electron micrographs with practically no change in grease structure.

Good results are obtained by a combination of the smear and aerogel methods. McClellan and Cortes,[55,56] for example, obtained very good photomicrographs by treating grease specimens smeared directly on a metal screen after Browning's method without using a film coating (Fig 6).

Embedding Technique[95]

There is a brief indication of this method of studying the structure of polymolecular soap films. Such films are applied to methacrylic ester slides. Then sample strips of the slide are exposed to osmium tetroxide (contrast inducer and fixing agent), embedded in the polymerizing plastic and sliced into thin sections (*ca* 500 Å). Treatment with osmium tetroxide gives a big effect in the case of unsaturated fatty acids, caused by the osmium attached to the double bonds. After certain modifications this method should be of considerable interest for studying the structure of the soap crystallites forming the dispersed phase of greases.

In concluding this review of the methods of preparing grease specimens for electron microscope structure studies special note should be made of the vital importance of shadowcasting the specimens. This greatly improves the contrast and definition of the image in almost all the versions of sample preparation methods. By shadowcasting at small angles, the heights of objects on the specimen carrier can be determined. Chromium mainly, but also gold,[10,38] palladium,[14,15] uranium,[40] and platinum are suitable metals for shadowing.

Grease structure examinations are usually made with a direct electron beam (light field image). In only one of the authors' studies[41] were electron photomicrographs of the structure of a Na-grease containing graphite obtained with dark field illumination (Fig 7). The dark particles in this figure are graphite. In some cases the use of a dark field of view is quite justified.

MAIN RESULTS OF ELECTRON MICROSCOPE GREASE STUDIES

A large number of papers have been published to date concerning investigations of the structure of almost all the presently known types of greases and of the influence of various factors on their structure.

Morphology of Grease Structure

Various soap and inorganic greases were studied extensively by Farrington[19] and Vinogradov *et al*.[41] Interesting data are given in other

papers.[17,24,29,55,65,66,81] These investigations established beyond doubt that the dispersed phase of soap greases in most cases consists of ribbon-, needle-, or rod-shaped soap crystallites.

In the case of calcium based greases (certain synthetic cup greases) thickened with soaps of synthetic higher fatty acids obtained by oxidizing paraffin, Jackson and Booser[65] and Sinitsyn *et al*[67] showed that their dispersed phase consists of petal-shaped particles. The structures of the dispersed phase of the calcium and sodium greases are based on unrefined synthetic fatty acids and cannot be determined by electron microscopy.

It is usually considered that aluminium greases cannot be studied in detail under the electron microscope. However, according to Hamm[40] and McClellan and Cortes[55] these greases have the fibrous structure characteristic of most soap greases.

Amott and McLennan[73] demonstrated that in lubricants based on so-called complex soaps the particles of the dispersed phase are similar in shape and size to the fibres of normal grease-making soaps.

Taking advantage of the results of an electron microscopic study of greases and determining the total surface area of the dispersed phase particles (by nitrogen absorption) in the dry structure skeleton of soap aerogels, Peterson and Bondi[31] and, later, other workers[58] showed the soap fibres to be solid continuous particles without pores.

The relatively large size of the solid hydrocarbon crystallites in hydrocarbon greases makes possible examination of their structure under the optical microscope. There are serious difficulties in using the electron microscope in this case owing to the poor contrast of the crystallites and the ease with which they are destroyed by the electron beam. The structure of such greases can be studied successfully by a method developed for investigating the crystals of individual *n*-paraffins.[14,15] The *n*-paraffins are usually crystallized out of solution in petroleum ether. A drop of a suspension of paraffin crystals is applied to the specimen carrier. To protect the delicate crystals from the action of the electron beam in the microscope and to bring out the details of their structure, it is advisable and often necessary to shadowcast the object with a sufficiently thick layer of metal (palladium,[14,30] chromium[67]).

The remarkable studies of Dawson and co-workers[14,15,30,35,83] not only demonstrated the possibility of obtaining contrasty electron photomicrographs of crystals of higher *n*-paraffins and *n*-alcohols, but also established the nature of their growth in the process of formation. It was found by Anderson[35] that the rhombic crystals of these compounds have a step-pyramidal structure (Fig 8). Crystal growth proceeds spirally from the centre of the rhomb (crystallization centre), the height of each step equalling the length of the hydrocarbon molecule.

Farrington[19] and Vinogradov *et al*[41] found that the dispersed phase of bentonite grease consisted of plate-like particles of laminated montmorillonite. The structural skeletons of silica gel and carbon black greases[19,66,81] and of greases thickened with polyvalent metal carbonates[62] are formed by chains of very tiny isometric particles. Those of phthalocyanine[28,71] and alkyl urea greases[75] consist of needle-like crystallites.

In greases with both inorganic and organic thickeners the primary particles of the dispersed phase are observed to agglomerate into porous formations. According to McClellan and Cortes,[55] the individual particles of inorganic thickeners do not intergrow with one another. Attapulgite[66] and the sodium, lithium, and barium salts of the methyl ester of *n*-octadecyl terephthalic acid[57] possess the fibrous structure characteristic of soap greases. Figs 9 and 10 show examples of electron micrographs of inorganic and organic thickeners.

Electron microscope studies have established that the structural skeletons of greases may be formed both by highly anisometric particles and by particles of symmetric shape. This is a conclusion of fundamental importance. It shows that the most important factor in the building up of the structural skeleton in such systems is not the shape of the dispersed-phase particles, but their size and ability to aggregate.

The Effect of Composition and Conditions of Preparation on Grease Structure

Many electron microscope studies have been made to correlate various factors with grease structure. Sproule and Pattenden[7] and Birdsall and Farrington[8] showed that water stabilizes calcium greases and promotes the formation of fibres 400 to 500 Å in diameter with a characteristic twisted shape (see Fig 2). Milling favours the formation of twisted fibres, but addition of calcium acetate and stearic acid promotes crystallization of the calcium soap as flat ribbons.

Bondi and co-workers,[18] as well as many others,[7,8,20,61] examined the effect of polar additives on the structure and properties of soap greases. Bondi investigated the effect of adding glycerin and certain polyesters to sodium greases and found that they promote the growth of the fibres. Petroleum resins and high molecular weight polymers, on the other hand, inhibit fibre growth in these greases.

Research on the effect of the alkalinity and acidity of the medium on the fibre structure has been reviewed previously.[85,94] Sinitsyn *et al* have investigated the structures formed by lithium stearate in spindle oil on cooling from 200°–210° C. In the presence of free alkali (up to 0·2 per cent NaOH) soap crystallites in greases are very small in size. The length of the ribbon- and needle-shaped crystallites may be dozens of times less in this case than in neutral greases.

Lithium soap fibres formed in acidic media (up to 3·5 mg KOH acid value) were found to differ little from those in neutral media, and similar results were found for sodium greases. Fig 11 illustrates the differences in the fibres formed in media of different alkalinity. Vamos and Guba[94] found similar effects with free alkali and higher fatty acids in calcium greases with complex soap thickeners.

The chemical composition, viscosity, and certain other characteristics of the dispersion media may have a definite effect on the properties and structures of greases.[10,18,25,67] A study of sodium greases[18] made with various petroleum oils showed that relative to the action of other factors the chemical composition of the dispersion medium usually affects grease structure little. Investigations of lithium greases by Hotten and Birdsall[25] and later by the present authors[67,68] showed, however, that the influence of the nature of the dispersion medium on structure in soap greases cannot be ignored. The prime factor is the viscosity of the dispersion medium. Increasing the viscosity to a certain limit promotes growth of the dispersed phase particles in Na-greases.[18] According to the authors' data,[68] raising the viscosity of the oil in lithium greases greatly increases the length-to-width ratio of the lithium soap fibres as shown in Fig 12.

The electron microscope was used successfully for studying the dependence of structure on the composition of the dispersed soap phase. The influence of the soap cation was studied when investigating the structure of various types of soap greases (based on Ca-, Na-, Li-, Al-, Ba-soaps, etc.). A very detailed study of calcium[10,18,33] and lithium greases[21,22,25] has been made of the influence of the structure and size of the radical in the soap molecules on the structure of the grease. Fig 13 shows the marked difference in structure between fibres of lithium stearate and lithium-12-hydroxy stearate. The effect of the anions and cations on the fibrous microstructure of pure soaps was studied by Hattiangdi,[9] who examined Li-, Na-, K-, Rb-, and Cs-palmitates, and the Na-salts of stearic and oleic acids.

The kinetics of structure formation in the process of grease production is a fruitful field for electron microscope research techniques. The influence of the conditions of manufacture on grease structure has been studied,[34,37] but this only in part. However, manufacturing conditions of lubricants have recently been controlled on the basis of an electron-microscope study of their structure.[88] More attention is being devoted to investigating the influence of the cooling rate on soap grease structure. In grease thickened with Li-oleate and stearate, lowering the cooling rate promotes the formation of well-formed, comparatively large fibres.[25] Similar results were obtained with Na-greases.[18] In work by Trapeznikov,[87] where the Li-stearate melt was cooled in petroleum oil in two stages, it was found that at a very high cooling rate broken fibres, as it were, are formed. Vold and co-authors[20] found that increasing the cooling rate promotes secondary aggregation of the (dispersed soap phase) particles in greases. Besides the cooling rate a definite influence on the structure of Li-grease is exerted by the maximum heating temperature,[21,22] as well as by the thermal[77] conditions of preparation.

A vivid idea of the effect of the cooling rate on the structure of greases is given by the electron photomicrograph shown in Fig 14. The system studied was low viscosity petroleum oil plus 10 per cent Na-stearate; the greases were all neutral, and were made at a wide range of cooling rates.

Yermilov[63] demonstrated the influence of thermal and ultrasonic methods of dispersing the thickener on the structure of Ca- and bentonite greases.

The Effect of Structure on Properties of Greases

The development of the electron microscope made possible an attempt to establish the relationship between the nature of the structural skeleton (size and shape of dispersed phase particles, etc.) and the bulk properties of greases. Papers by Moore and Cravath,[17] Bondi and co-workers,[11,18] and others[39,43] show that the consistency of greases increases (*i.e.* the penetration decreases) with increasing fibre length/diameter ratio. It is interesting to note that a similar dependence was obtained for oil thickened with nylon fibres approaching large soap fibres in size.[11] Hotten and Birdsall[25,27,42] established a relation between the penetration of greases and the surface-to-volume ($s : v$) ratio of the particles. Raising $s : v$ from 10 to 50 microns^{-1} lowers the penetration of the greases. The same authors confirm Farrington's conclusion[19] that increasing the size of the primary particles forming the dispersed phase of lithium greases increases the granularity of their texture and lowers the transparency of the grease.

Panzer[90] found that as the particle size of the soap decreased, the viscosity of complex-soap Ca-greases rises and their viscosity anomaly grows. Attempts have been made to relate data from an electron-microscope investigation to the elastic-viscous properties of greases.[91] Some data increasing the influence of grease structure on rheological properties can be found also in a paper by Rossi and Quarleri.[46]

Direct observations from electron micrographs have been reported,[21,41] showing that soap fibres in greases become oriented in the direction of shear during flow. These data agree well with evidence from other studies on grease flow by optical polarization, dielectric permeability, and X-ray methods.

Bondi and co-authors[11] demonstrated that the colloidal stability of soap greases improves if the dispersion of the thickener particles is increased. Rochow[40] finds that increasing fibre length in alu-

minium greases improves the colloidal stability. Borg and Leet,[59,89] however, find that increase in length/diameter ratio of fibres in lithium greases reduces the colloidal stability. Panzer,[90] working with calcium greases, found a similar effect.

Borg and Leet[59,89] also reported that increasing length/diameter ratio of lithium soap fibres resulted in improved mechanical stability of the greases, and less liability to thixotropic restoration after breakdown. This was not confirmed by Renshaw,[45] who considered that the mechanical properties are affected much more by the nature of the forces between the particles comprising the structural skeleton. A similar view is put forward by Vold,[54] who considers that the effects of particle size and shape may be secondary to the conditions of aggregation in determining the mechanical properties of greases.

The Effect of External Influences on Grease Structure

Many papers have been devoted to the observation of structural changes after mechanical action. It should be pointed out that electron microscopy makes it possible to estimate the size and shape of primary dispersed-phase particles, but not the nature of their interaction, *i.e.* the nature of junctions between fibres. That is why practically all published investigations in this field have been concerned with changes in shape and dispersion of soap fibres.

The first paper demonstrating the effect of mechanical influences on grease structure was published by Bondi *et al.*[11] Marusov[16] studied the changes in structure of sodium greases occurring after repeated passage through a capillary viscometer. However, Marusov formed no definite conclusion on the effect of such treatment on the size of the soap fibres. Moore, Cravath, and their co-workers studied the breakdown of the structure of various greases in a Shell roll tester.[11,17,18] They showed that when greases break down their soap fibres decrease mainly in length. Only after prolonged treatment are the fibres of sodium greases observed to split axially. The same study showed a relationship between the decrease in size of the soap particles and the consistency of the grease. Brown and his co-workers[21,22] broke down lithium greases using a penetration worker, a colloid mill, and a Shell roll tester. None of the laboratory methods could break down the grease structure as intensively as when the grease is used in rolling bearings,[21] where the soap particles break down so intensively in as little as 20 minutes[21] that under low electron microscopy magnifications (tens of thousands of times) their shape and size cannot be determined. Brown's work[21,22] suggested that soap fibres are broken down as a result of mechanical rather than thermal influences (local temperature flashes and overheating in the bearings).

In more recent papers[27,36,39,43,45–50,52,61,76–78,92,93] involving the use of the electron microscope for studying the mechanical stability of greases, the structural changes are followed during tests in laboratory apparatus (four-ball machines, etc.) as well as under various practical conditions of use (in railway axle boxes, rolling bearings, etc.).

Soap fibres in lithium greases have been observed to agglomerate as a result of mechanical and thermal influences.[45–50,76,78,79]

The only case where the microscope has been used to study the mechanical stability of inorganic greases is an investigation by Peterson and his co-workers.[53] It was found that mechanical treatment of inorganic greases does not break down the dispersed-phase particles, but leads to their agglomeration.

A fundamental question was solved by Bondi *et al*[18] and Leet.[39–43] Prior to this work it was not known whether or not soap thickeners re-crystallize when worked greases are allowed to rest. The data given in the above papers, including the electron photomicrographs, gave clear proof that the soap fibres of various types of greases increase in size upon resting. Vold[20] made a study of structural and other changes in calcium stearate–cetane gels stored for two years. He found that besides changing its structure, the system softened and its colloidal stability was reduced.

Sinitsyn, Leontyev, and Klimov studied the degradation of calcium, sodium, and lithium greases at temperatures from 10° to +100° C and at shear rates from 1 to $2{\cdot}5 \times 10^5$ sec^{-1}. A study was also made of the effect of a long rest (up to one year) on the structure of the sheared greases. The most drastic structure breakdown was observed in the case of cup greases. Fig 15 shows the dispersed-phase of a cup grease before breakdown, and after it had been held subjected to a shear of $2{\cdot}5 \times 10^4$ sec^{-1} for several seconds. The soap fibres of Na- and Li-greases were not completely broken down at the highest shear rates. They retained their size and shape, which were easily determined by electron photomicrographs at magnifications of the order of $10^4\times$, as illustrated by the photographs in Fig 16.

In calcium greases the particles break up mainly at right angles to the fibre axis; in sodium greases they split parallel to the fibre axis and break at right angles to it. Lithium soap fibres are observed to become shorter and the broken particles agglomerate. Thus a study of the process of grease breakdown with a very wide range of shear rates confirmed the earlier findings.[11,17,45,76]

The effect of temperature on the structural breakdown of Na-greases at various constant shear rates has also been studied. Fig 17 shows that destruction of the soap particles decreases with rising temperature.

Broken-down greases studied by Sinitsyn, Leontyev, and Klimov showed no substantial change in particle size or shape upon resting. Fig 18 shows electron photomicrographs of a Na-grease broken

down at a shear rate of $2{\cdot}5 \times 10^5$ sec^{-1} and of the same grease after a year's rest. A comparison of these micrographs with those reported previously[18,39,43] suggests that the change in structure during resting of the greases depends essentially on the nature of the soap particles.

There is a qualitative correlation between the rheological properties of greases and the size of the soap particles is revealed by electron microscopy. Smaller particles in the greases are associated with lower ultimate shearing strength and viscosity. This makes it doubtful whether the ideas put forward by Panzer[90] are of general application.

Electron microscope studies have helped to establish a relationship between behaviour under conditions of boundary lubrication and the structure of greases.[76] In this work it was found that changes in the coefficient of friction resulted from changes in grease structure.

Very little material has been published on the effects of non-mechanical influences on grease structure. However, Brown and co-workers[21] established that oxidation of lithium greases causes the soap fibres to break. The same effect was found by Kabes[51] for sodium greases. Carroll and Calish[60] and Hotten and Carroll[80] have shown that the effect of large γ-ray doses on sodium greases is to produce a considerable breakdown of structure (Fig 19).

CONCLUSION

During the 15 years which have passed since the first electron photomicrographs of greases were obtained, considerable work has been done in this field and substantial achievements have been made. This period has been largely associated with test work and purely qualitative results. To-day, however, electron microscopy has become the chief method of determining grease structure and controlling the effect of various factors on it. This trend, represented by a rather large number of publications, will evidently continue to develop intensively.

Two questions await detailed investigation:

1. Examination of the statistics of size distribution of the dispersed-phase particles. Without this it is impossible, first, to estimate the poly-dispersity of greases and its effect on their properties, and secondly, it is difficult to determine reliably enough the effect of various influences and resting of the greases on their structure. Up to now all the conclusions with respect to this question have been of a purely qualitative nature.

2. Insufficient attention has been devoted to the study of the process of dispersed-phase particle formation in greases, the structure of the particle surfaces, and the nature of their aggregation. The process of structure formation in homogenized greases must depend substantially on the nature, state, and structure of the surface of the dispersed-phase particles.

Solution of the above indicated tasks involves very laborious or delicate investigations. However, without these it will hardly be possible to make any considerable progress in the further study of grease with the aid of the electron microscope.

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It has been drawn to the attention of the Institute that in publishing the paper entitled "Queen Elizabeth Islands of Arctic Canada and their Petroleum Prospects" by A. Bryce Cameron, in the May 1961 edition of the *Journal*, certain credits for material emanating from publication by the Department of Mines and Technical Surveys, Ottawa were omitted.

The list of credits and acknowledgments should have included:

1. Figs 19 and 21 were reproduced, with minor changes, from Black, W. A., "A Report on Sea Ice Conditions in the Eastern Arctic, Summer 1958," Geographical Paper No. 20, Geographical Branch, Department of Mines and Technical Surveys, Ottawa, 1959.

2. Fig 4, (Explorations), after Plate No. 1, Atlas of Canada, Geographical Branch, Department of Mines and Technical Surveys, Ottawa, 1957.

3. Figs 17 and 18, being Figs 7 and 8 in: "Pilot of Arctic Canada," Vol I, First Edition, issued by the Canadian Hydrographic Service, Department of Mines and Technical Surveys, Ottawa, 1959.

The Institute wishes to express regret to both the author, A. Bryce Cameron, and to the Department of Mines and Technical Surveys, Ottawa, for these omissions.

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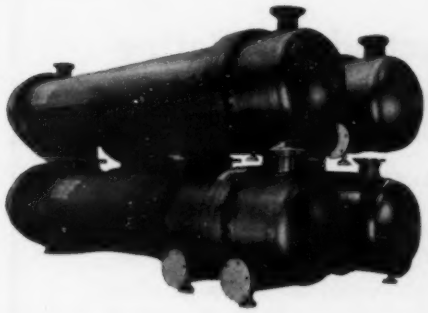

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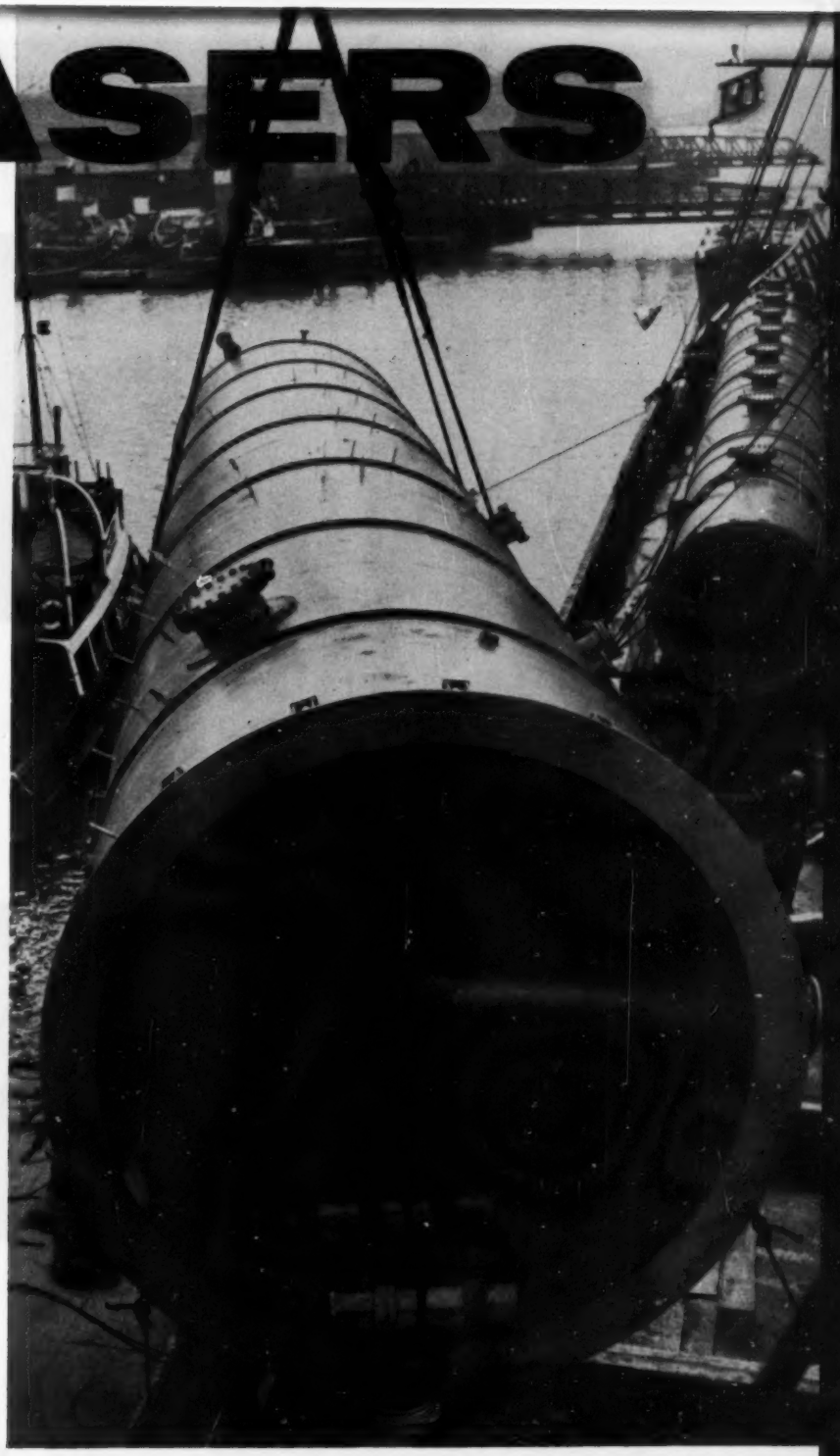

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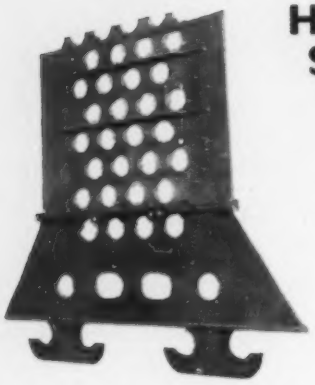

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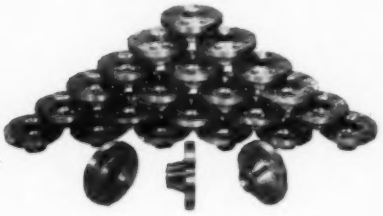

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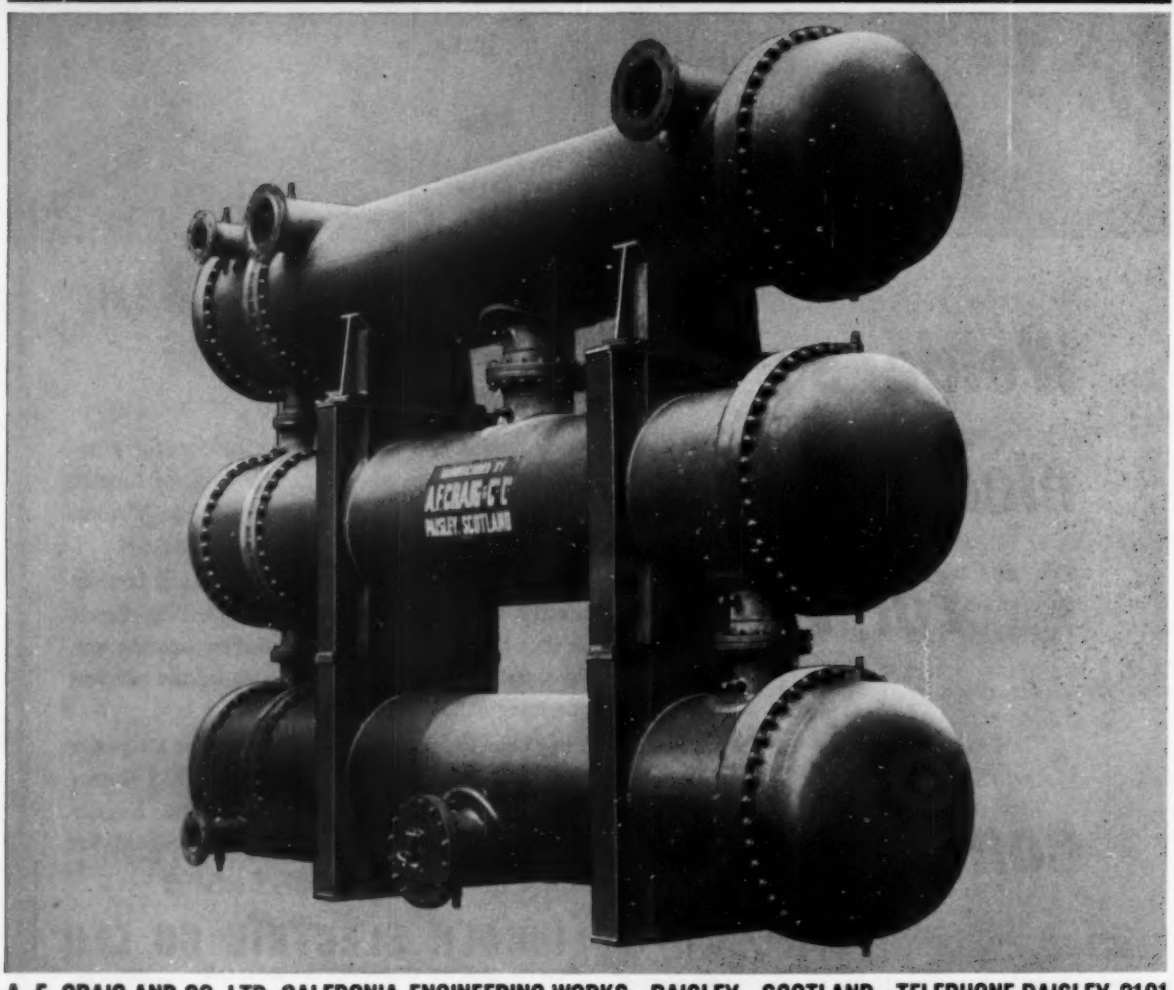

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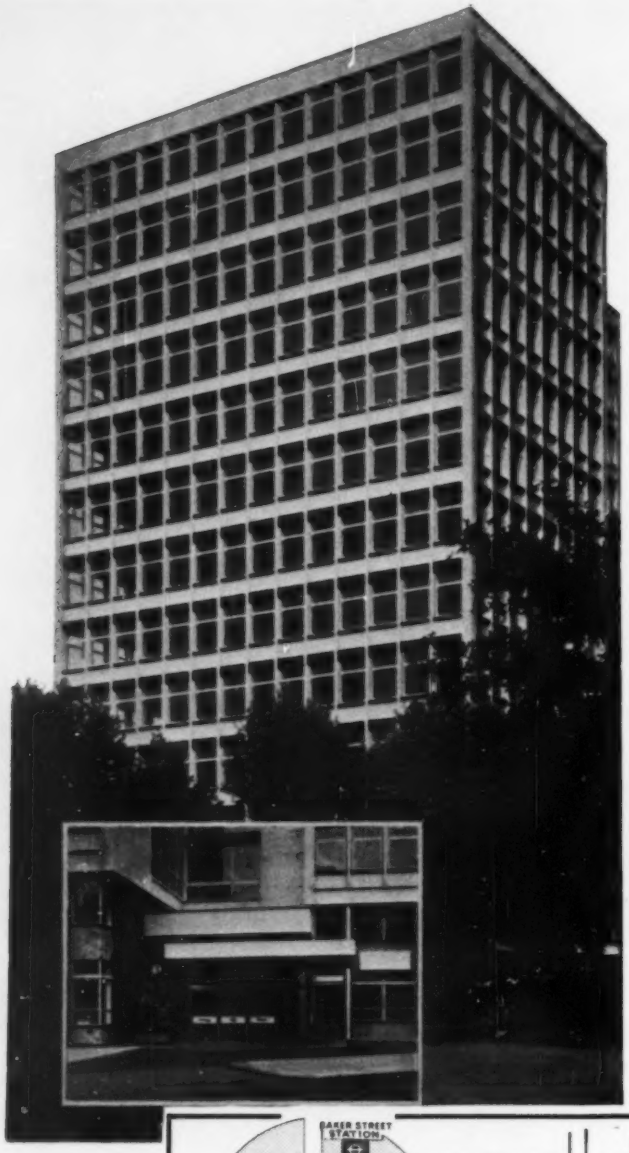

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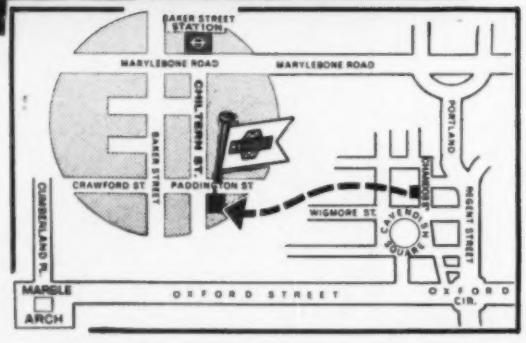

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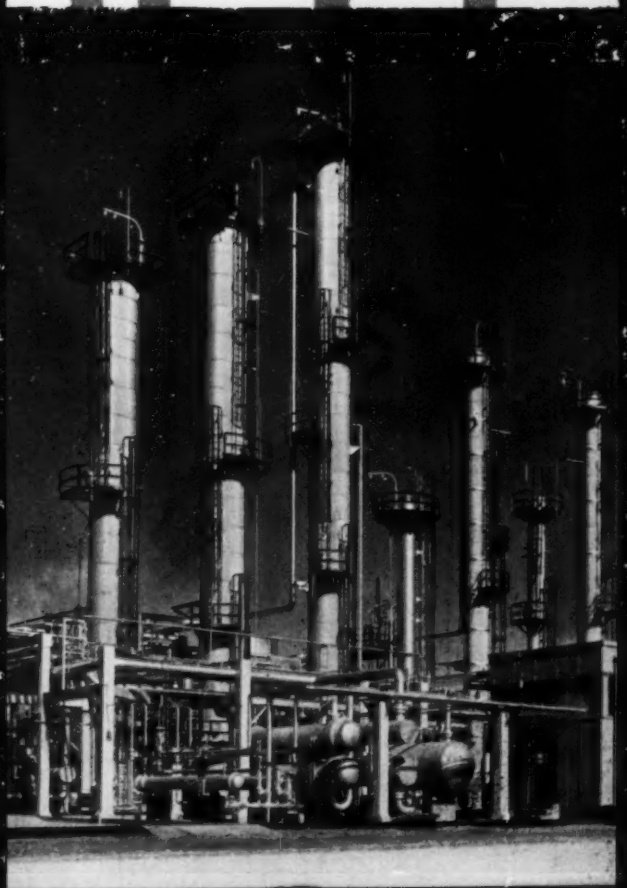

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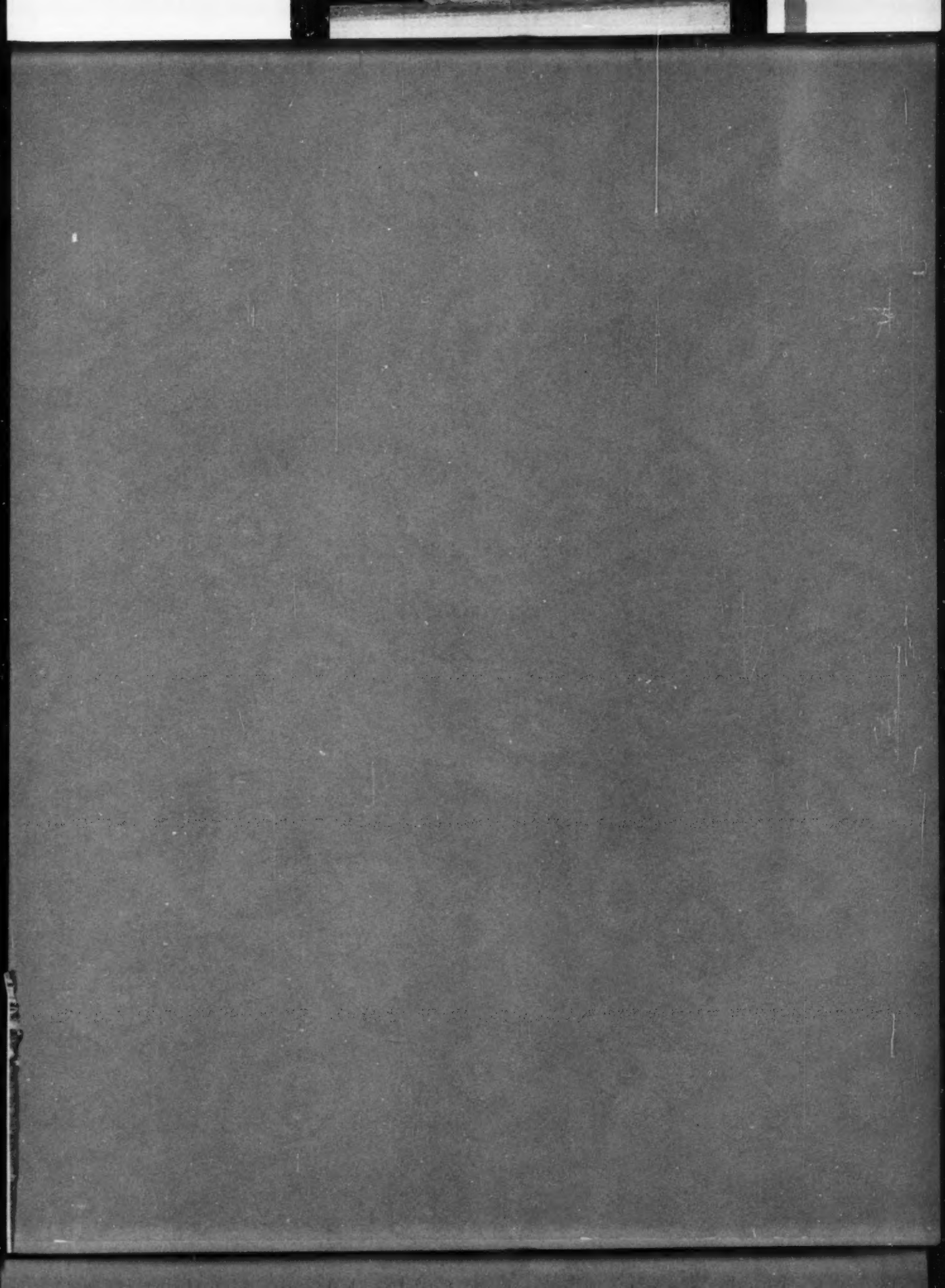
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